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QUARTERLY PROGRESS REPORT

ON

LABORATORY INVESTIGATIONS AND RESEARCH

TOWARD THE DEVELOPMENT OF

NICKEL CADMIUM ALKALINE STORAGE BATTERIES

31 DECEMBER 1947

- Contract W-36-039 SC-32271 -  
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NICKEL CADMIUM BATTERY CORPORATION  
EASTHAMPTON, MASSACHUSETTS

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QUARTERLY PROGRESS REPORT.

1 Oct. - 31 Dec 47

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LABORATORY INVESTIGATIONS AND RESEARCH

TOWARD THE DEVELOPMENT OF  
NICKEL CADMIUM ALKALINE STORAGE BATTERIES.

By

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ARTHUR FLEISCHER, ~~PH.D.~~

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Contract W-36-039-SC-32271

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Vice Pres. and Chief Engineer

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NICKEL CADMIUM BATTERY CORPORATION

EASTHAMPTON, MASSACHUSETTS

LABORATORY INVESTIGATIONS AND RESEARCH TOWARD THE  
DEVELOPMENT OF NICKEL CADMIUM ALKALINE STORAGE BATTERIES

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LABORATORY INVESTIGATIONS AND RESEARCH TOWARDS THE DEVELOPMENT  
OF NICKEL CADMIUM ALKALINE STORAGE BATTERIES

1. FOREWORD

This progress report, the second following the annual report of 30 June 1947 covers the work of the period, Oct. 1, 1947 to Dec. 31, 1947. The work of this period has carried forward the experimental survey and has resulted in the accumulation of a large mass of data relating to the science and technology of sintered plates. It has carried us forward to the assembly of sintered plate batteries whose performance and life will be determined during the remainder of the contract. Our present knowledge, reflected by the experimental data which has been presented prior to this report and in the present report, will enable the construction of the required all sintered plate batteries. It has pointed to many means of improving the batteries and the experiments have in many cases defined the phenomenon occurring in alkaline batteries.

The program, under the contract, is intended to culminate in the manufacture of between 20 and 200 alkaline-type batteries with sintered positive and negative plates, with the batteries to meet the requirements of specification FL5086, 3/17/46. It now appears that there is a marked increase in interest in performances at high rates of discharge and this may to a certain extent change the exact course of the program. The experimental work has indicated up to this time that for batteries with sintered plates and performances in the ordinary ranges of 5, 8 and 20 hour rates can be carried by plates of either 2 or 4 mm. of thickness. The requirements up to this time have led us to concentrate on the thicker plates since the evidence from the experimental work showed a decided advantage in favor of the thicker plates from several points of view.

The present report does not present a complete analysis of the data amassed up to this time. In proceeding with the program, there must be a constant thought of correlating what is done in the production of sintered plaques with the ultimate behaviour of a plate in the battery and the properties of the product of the initial step must be evaluated in terms of the final use. It is a natural consequence, therefore, that ideas will be promulgated and transferred into actual experiments, often before the guiding principles have been thoroughly established. It is to be expected that these progress reports will often represent a stage in the evolution of a correct interpretation or what might be embodied in

## 1. Foreword (Contd.)

the expression of "thinking aloud." There is every intention of presenting thorough and logical analyses of the accumulated data, though many of the developments will supply seed for the desired further development of highly efficient and rugged sintered plate alkaline batteries.

It has been indicated that the work under this developmental contract has provided the background of fulfilling the specifications of the battery on which it was based. The accumulation of data has been made from the point of view of providing the necessary information on which a plant for quantity production can be designed. The importance of this background of information can not be overemphasized when one analyses the difficulties encountered in the manufacture of sintered plate batteries by A.F.A.G. There is an overwhelming tendency to assume that the translation from laboratory operations to industrial manufacturing was made with due disrespect and disregard for the detailed factual information which was developed and which seems to have been destroyed in the wake of the American invasion of Germany. In any event, the German experience should teach the need for translating each step in terms of the accumulated laboratory experience.

The corollary of the proposition that the translation of design from laboratory to production unit be made on the basis of experimental data, is embodied in the principle that adequate means of controlling the unit processes be provided. Need for this step is clearly apparent from our studies and experiences with light carbonyl powder. We now have a definite picture of the requirements in the matter of the required control and a greater effort should be devoted to the study of non-destructive methods of testing. The need for such control in the present instance may be accentuated by the use of batch processing, yet reference to the technique and results of the Durac sintering operation is a forceful reminder that the success of a large scale production may well be a function of the methods of control applied to the manufacturing processes.

These considerations apply to the expansion of the operations to the manufacturing scale. In the same way it is possible to project the present findings into the future possibilities of research. The results of fundamental investigations can not always be predicted, but it seems a safe bet to assume that a better understanding of the nature of the nickel-cadmium cell and the mechanism of the processes will lead to new

## 1. Foreword (Contd.)

and improved batteries. It is a virgin field from the point of view of the application of the newer tools and theory of modern science.

As an example, the mystery surrounding the function of lithium ion in the alkaline cell should not continue to baffle us and remain a purely empirical phenomenon. The clues are in the chemical and physical characteristics of the lithium ion and its interaction with other components of the system. If the effect is purely one of diffusion and the related hydrodynamical effects of lithium ion, the vast field of organic cations should yield one of characteristics reproducing the effect of the lithium ion. This would establish a wedge into the closed book of explaining the action in reasonable terms and then predicting the course of events under less favorable conditions and thus opening the road to improvement.

Or, let us look into the mechanism of the vacuum impregnation process. To date we assume that each successive vacuum impregnation of nickel plaques precipitates lesser amounts of active masses in the pores of the plaque because of some difficulty in reaching all of the pores with solution, despite the vacuum treatment. The experiments with porous graphite plates gives some credence to this explanation provided that it is assumed that the pores are smaller than a certain size. On the other hand, we know little about the possibilities of a resolution of previously precipitated active mass in the strong nickel and cadmium nitrate solutions, in which range soluble basic compounds can exist even if only in a metastable state. Here we might easily visualize the application of isotope tracer technique to evaluate the chemistry of the process.

Illustrations may be added up to a large number. The present work is concerned in examining such possibilities for the future possibilities for development and expansion of an item like the nickel-cadmium battery are often closely linked with an intelligent appraisal of the field of research and "room" for improvement. There is a certainty that the field is rich in its possible development. It may be likened to the favorable geological indications for tabulating "probable" ore reserves, awaiting development and mining.

## 2. PURPOSE

This progress report covering the sixth quarter under the contract gives a resume of the work on sintering light carbonyl nickel powder to produce porous plaques, the impregnation of the porous plaques with nickel or cadmium nitrate solutions to produce positive or negative plates, and on the testing of such plates in cells to determine capacity and behaviour. The work continues along certain lines of exploration but sufficient experimental data is presented to show that a satisfactory state of progress has been reached with regard to the objectives of the program and the contract.

## 3. SUMMARY

a. A total of 383 plaques were sintered of which 130 were S plaques, 10.5 sq. in. in area, prepared in the 3.6 x 3.0 inch two-piece form and of which the balance were prepared in the three-piece FM forms, mainly with an area of 14 sq. in. The compacts were assembled in the manner previously described and sintered under the usual conditions in a protective gas atmosphere.

b. The plaques were made from light carbonyl powder using a series of 5 lbs. samples, labeled B-1 to B-10, received from International Nickel Co., and from the first large batch of 1000 lbs. received from this company. These powders showed a variation in apparent density, determined with the Scott volumeter, from 0.46 to 0.88. The apparent density of the powder packed into the form, corrected for the grid weight and volume, was higher by an average ratio of 1.37. This increase in apparent density with the gentle handling of the powder in making the compacts may be an important factor in controlling the percentage shrinkage on sintering.

c. The percentage shrinkage in thickness on sintering of the light carbonyl nickel powders has shown a steady decrease in the series of powders which have been made available. Expressed in terms of the ratio of form or compact thickness to final plaque thickness, the shrinkage has varied from 1.48 to 1.23. This approaches the figure reported in German practice, although one German nickel powder showed an appreciably lower shrinkage. For a given powder, such as International Nickel Co's B series, it appears that shrinkage in thickness on sintering of compacts decreases with increasing apparent density.

d. The B series of powders has the bulk, 92 to 95%, of its particles in sizes smaller than 325 mesh (44 microns). The aggregate or effective particle size and shape appears to be a function of treatment and environment, carrying the work into a field where quantitative relationships are complex and ill-defined at the moment.

### 3. Summary (Contd.)

e. Plaques of increased area have been made by increasing principally the height of the plaque originally designed for the FMSL 5086 battery, to permit an increase in capacity of the battery. The dimensions had been based on prior experience with pocket-type nickel cadmium alkaline batteries.

f. Change from the two-piece to the three-piece form for sintering compacts resulted in an appreciable increase in the weight of the form thereby necessitating a change in sintering conditions to produce plaques of the desired thickness. Auxiliary physical tests such as hardness or electrical resistance, particularly non-destructive testing, is desirable to permit exact comparison of plaques of different dimensions.

g. The effect of time of sintering at one minute intervals from 3 to 15 minutes was studied for the two forms. The shrinkage curves were parallel and did not reach a steady state within the time limit proscribed for these tests.

h. Experiments with thin rolled and electroformed nickel sheet grids indicate that satisfactory plaques of the required porosity and flatness can be made by allowing a sufficient space for expansion of the grid in the form cavity. This item appears to be of importance in the design of grids without a re-enforcing heavy frame although on the latter case it is postulated that the failure to allow for expansion of the frame during the sintering process may well be responsible for residual stress which exerts its effect in the subsequent electrochemical behaviour.

i. Experiments with thin pre-sintered carbonyl nickel grids failed in providing an improved degree of adhesion between the sintered powder and the grid. The absence of any tendency for carbonyl nickel powder to adhere to prepared surfaces is in marked contrast to experiences in the decomposition of nickel carbonyl.

j. The origin of cracks in plaques, made evident on sintering compacts of carbonyl nickel powder, are discussed. The cracks are apparently of little or no effect on the ultimate electrochemical characteristics of the finished plates.

k. No further work has been carried on with copper powders since the accumulated evidence favors the possibility that difficulties encountered in the German negative plates are a result of the use of copper in the negative plates.

l. Impregnation of plaques with cadmium nitrate solution to produce negative plates has been shown to result in approximately the same loading at atmospheric pressure as by the multiple vacuum procedure. The weight gain in the first impregnation cycle by the atmospheric procedure exceeds the weight gain for a similar vacuum impregnation.

### 3. Summary (Contd.)

m. For the normal solution concentrations, increase in the temperature at which atmospheric impregnation is made, decreases the weight gain for both positive and negative plates.

n. The impregnation process is characterized by a recurrent cycle of gas evolution under the conditions of atmospheric impregnation. This phenomenon probably accounts for the variation in weight gain as a function of the immersion period.

o. Impregnation of plaques with nickel nitrate solution to produce positive plates at atmospheric pressure results in a higher weight gain in a single cycle for periods of one hour or more than in the corresponding vacuum treatment. The small irregular increase in weight gain with increased time beyond one hour offers no advantage judging solely by theoretical capacities. Multiple cycles of impregnation at atmospheric pressure result in theoretical capacities of the same order of magnitude as obtained with multiple vacuum impregnations.

p. Maintenance of constant acidity during the impregnation process at atmospheric pressure does not enhance the theoretical capacity, and actually showed a slight decrease over comparable tests in which the initial acidity was allowed to change naturally.

q. Vacuum impregnations for multiple cycles shows higher theoretical capacities for the negative plates over positive plates, of the order of magnitude of 2:1. On four cycles of impregnation, the positive plates show that about 31% of the pore volume of the plaques is occupied by active mass.

r. Multiple vacuum impregnations of a series of plates sintered for times varying from 5 to 15 minutes showed a constant theoretical capacity based on pore volume, namely 1.27 grams of nickelous hydroxide per cubic centimeter of pore volume,

s. Multiple vacuum impregnation of macroporous graphite plates with pore diameters varying from 33 to 140 microns showed an approximately constant gain per impregnation for plates with pore diameters of 48 microns or over. The plate with a pore diameter of 33 microns showed a continual sharp decrease in theoretical capacity in successive impregnations. The net gain in theoretical capacity varied and was less than with nickel plates.

### 3. Summary (Contd.)

t. Experiments were carried out using lithium hydroxide electrolyte for the cathodic polarization of both positive and negative plates. There was no abnormal increase in weight showing the absence of a direct combination of lithium ion with the active masses.

u. Pore volumes calculated from the volume of active mass and the moisture content after each impregnation were found to be slightly lower than the values calculated from measurements of the plaques. The discrepancy may be due to the difference in density of the precipitated mass from that of the crystalline hydroxides.

v. The testing of individual plates for electrical capacity is discussed and the inauguration of a program for changing the testing procedure is shown to be in order. Variations in capacity found in a series of tests of a given plate are noted in tests on cells made up of pocket type positive and negative plates.

w. Positive plates impregnated in a single cycle in strong nickel nitrate solution at elevated temperature and atmospheric pressure showed capacities close to the value reported for Durac positive plates and gives rise to the possibilities of developing a single step impregnation procedure.

x. Multiple atmospheric impregnation of positive and negative plaques at room temperature yields plates which show capacities at coefficients of utilization approximately the same as for plates prepared by multiple vacuum impregnation. The capacities found for the plates prepared by multiple atmospheric impregnations are slightly lower than the plates prepared by multiple vacuum impregnations. The relationships permit the evaluation of impregnation processes on the basis of theoretical capacities.

#### 4. DISCUSSION:

##### a. Sintering

(1) Light Carbonyl Nickel Powder. Carbonyl nickel powders, produced by the thermal decomposition of gaseous nickel carbonyl and consisting substantially of pure nickel metal, may be subdivided into two classifications. These classifications have been conveniently referred to as heavy and light in deference to the determinations of the apparent (bulk) density which has been the principal method of characterization of the powders. The heavy powders fall into a range of densities from 2 to 2.5 and yield plaques, on sintering for 10 minutes at 1500°F-1800°F, having a porosity of about 65% by volume. The light powders fall into a range of densities from 0.5 to 1.1 and yield plaques, sintered under conditions similar to the above-mentioned conditions, having a porosity of 75 to 85% by volume. The inherent advantage in weight saving by using the light powder is so great and so important that work has been concentrated on this type of powder.

The light powder for this program, except for several very small samples, has originated in England and has until recently with the receipt of a 1,000 lb. batch, consisted of small shipments aggregating up to 15 lbs. but generally designated with batch numbers for each 5 lbs. The light carbonyl powders which have been received to date have varied in apparent density. This property has been determined for all of the powders with a Scott Volumeter, an apparatus used for the determination of the apparent density of pigments used in the paint industry (7a). This method yields an arbitrary but reproducible figure which is lower than the apparent density of the powder in the form, calculated from the weight of powder, volume of the form, and corrected for the volume and weight of the grid. The ratio of these two apparent densities is found to be from 1.3 to 1.45. The data have been assembled in Table No. 1.

The variation in the apparent densities of the light carbonyl powders which have been described in Table No. 1 does not reflect any easily detected difference in the capability for sintering. From all superficial appearances, the plaques produced are entirely satisfactory and possess a porosity from 75 to 85%, depending mainly on the weight and volume of the grid. The variation, however, poses a problem for a manufacturing process, when the various factors are extrapolated to the usual expected practice in American methods of mechanisation.



4. Discussion: a. Sintering (Contd.)

Table No. 1  
Light Carbonyl Nickel Powders  
Description & Apparent Densities

Powder No.	Description	Amount Received lbs.	Apparent Density g/cc		Ratio
			Scott Volumeter	In Forms	
P-10	I.G.Farben A.G. (Fort Monmouth)	small	0.85	nd	
P-12	Inco KIG- AFA type	small	0.59	0.82(1)	1.39
P-13	" KIG-2	"	nd	0.87(1)	
P-15	" KIG-3	3/4	0.62	0.88(1)	1.42
P-17	" KIG-2	small	0.63	0.85(2)	1.35
P-19	" KIG- AFA type	3 *	0.66	0.89(2)	1.35
P-21	" B-1	5 *	(0.53	(0.71	1.34
"	" B-2	5 *	(	(	
P-21-1	" B-3	5 *	0.67	0.80	1.20
P-21-2	" B-4	5 *	0.50	nd	
"	" B-7	5 *	0.48	nd	
"	" B-8	5 *	0.48	nd	
"	" B-9	5 *	0.46	nd	
P-21-3	" B-10	5 *	0.64	0.89	1.39
P-22	I.G.Farben A.G. (Carl Berg) 1932	small	0.92	nd	
P-23-1	Inco B-85, representative of 1000 lbs.		0.79	nd	
P-23-2	" " Cut Sample	250#	0.78	1.06	1.36
"	" " Grab "	"	0.88	nd	
P-23-5	" " " "	"	0.88	"	
P-23-6	" " " "	"	0.73	"	
P-23-7	" " " "	"	0.70	"	

Notes \* All these samples screened through 200 mesh (dry)  
(1) Table No. 1, p. 10 Ref 7(b)  
(2) " " " p. 6 " 7(c)  
(3) Values of apparent density by Scott Volumeter for P-19 and P-21-2 (B-7) were checked to within 0.01 unit by two independent laboratories.

#### 4. Discussion: a. Sintering (Contd.)

In the present studies, the procedure of making a compact, as previously described in the Progress Reports, consists in filling a graphite form with powder and a grid. The form consists of two or three graphite parts, but in either case the thickness of the form is fixed. It has also been shown that the use of a grid restricts the principal shrinkage on sintering of the compact to the dimension of the thickness. The production of plaques from these various nickel powders has shown a variation in the shrinkage in thickness. This is clearly illustrated by the data of Table No. 2; the first five plates represent samples taken from the 1000 lb. batch of powder while the other figures are representative averages based on the data presented in Tables I-XIII. The values for the shrinkage have been expressed in terms of the ratio, form thickness to plate thickness, which is the desirable figure for design purposes.

A variation in the shrinkage creates the problem of correcting for this property in the handling of various lots of powder unless the eventual powder manufacturing process can be controlled within the desired limits. At the moment, the safe approach will be to assume that limits of variation will be of the order of magnitude indicated in the table, and to consider the possibilities by which the process can be brought within the tolerances required for plates, with due consideration to their final assembly into batteries. There are a number of simple methods, such as shaping the plaque before or after impregnation, adjustment of the spacer piece of the mold for various powder characteristics, and blending of relatively large batches on a laboratory-controlled schedule. Each approach has its merits and any final decision would be determined to a large extent by considerations of cost.

It is apparent from the overall picture that the shrinkage in thickness of the carbonyl nickel powder has been gradually diminishing for like preparations and conditions, and has been approaching the value of 1.20 calculated from the data on German practice (Ref 7(d)). The Germans had apparently succeeded in preparing powders of even lower shrinkage, judging by the behaviour of sample P-22 (Table No. 2, VII) which represents a powder given to Mr. C. Berg in 1932 by officials of the I. G. Farbenind A.G. The important problem now reduces to an evaluation of the property which controls the shrinkage. The theory of powder metallurgy directs primary attention to particle size, distribution of particle size, ultimate

4. Discussion: a. Sintering (Contd.)

Table No. 2

Shrinkage in Thickness of Various Powders

Powder No.	Apparent Density			Shrinkage Ratio, Form Thickness: Plaque Thickness
	Scott Volumeter	$\frac{g}{cc}$ In Form	Ratio	
P23-1	0.79	1.14	1.44	1.34
P23-2	0.88	1.12	1.28	1.23
P23-5	0.88	1.12	1.28	1.29
P23-6	0.73	0.99	1.36	1.29
P23-7	0.70	0.94	1.34	1.30
P21	0.53	0.71	1.34	1.48
P21-1	0.67	0.80	1.20	1.42
P21-2	0.50			1.43
P22	0.92			1.05

#### 4. Discussion: a. Sintering (Contd.)

particle shape, and aggregate shape. Available information indicates that the particle size range measured by the accepted methods (sedimentation, electron microscopy, etc.) will be in the subsieve sizes. That the bulk of the light carbonyl nickel powders lies in this range is confirmed by the screen analyses of Table No. 3. In addition, several tests have shown from 2.0 to 2.8% by weight of the total to be minus 200 plus 325 mesh, so that from 92 to 95% of the powder can be considered as being minus 325 mesh in size.

It would appear to be self-evident that the range of properties to be determined has passed to the realm of instrumental analysis, requiring research with devices beyond the range of the usual developmental departments. An intense exploration is highly desirable so that the fundamental facts are clearly defined and thinking is properly oriented. Several observations on the qualitative behaviour of carbonyl nickel powder would indicate that the extension of research will involve some fundamental studies, concerning which our present day science has not yet clearly defined the active forces and their mode of action.

When nickel powder is moved and levelled gently in a form, it is clear from the figures in Table No. 1 that there is an increase in the packing which appears to be about 35% greater than that attained in a free fall. It is extremely difficult to visualize this effect simply on the basis of a difference in the arrangement of particles except for the postulation of a tendency for the particles to bridge and arch on impact from a free fall and the ready breaking-up of such bridges by even gentle levelling. If this is the case, the experimental procedure should be concentrated on the mechanics of packing and working powder into a compact.

It has also been observed that green compacts tend to form cracks when the form is jarred in any manner. An irregular line of division forms; this line may be microscopic in its breadth and barely visible along its length to the eye. There appears to be no satisfactory mechanical means for repairing such an incipient crack which increases in width on sintering and when such a crack has been suspected by the operator, it has been found necessary to empty and re-pack the form with powder to eliminate the crack.

4. Discussion: a. Sintering (Contd.)

Table No. 3

Screen Analyses of Light Carbonyl Nickel Powder

Powder No.	Description	Screen Analysis				
		Plus 80 mesh	Minus 80 Plus 200		Minus 200	
		Ind %	Ind %	Cum %	Ind %	Cum %
P21		0.2	0.4	0.6	99.4	100.0
P21-2	B4	0.13	0.9	1.0	99.0	100.0
"	B7	0.11	1.4	1.5	98.5	100.0
"	B8	0.15	1.1	1.2	98.8	100.0
"	B9	0.14	1.6	1.7	98.3	100.0
P21-3	B10	0.15	1.4	1.5	98.5	100.0
P23-2	Cut Sample	0.30	1.1	1.4	98.6	100.0

#### 4. Discussion: a. Sintering (Contd.)

On the other hand, these effects may also be inherent to the particular combination of aggregate sizes and shapes, which are actually produced in transferring the powder to the form. In the case of the present method of handling the powder, gas (residual carbon monoxide, air) may be considered as the dispersing medium. The effect of adsorbed films in the case of carbonyl nickel powder has been demonstrated in the range of temperature from 200° to 300°C (Ref. 7 e) but not at ordinary temperatures, and no work is available to explain its role at ordinary temperatures in processes such as compact assembly by methods similar to the one in use. When carbonyl nickel powder is suspended in water, it acts as a powder with a particle size much greater than has been established by electron photomicrographs, and to date no wetting agent has been found which disperses the aggregates and thus reduces the tendency of the nickel particles to form aggregates. Thus, the matter of the actual particle size condition of carbonyl nickel powder becomes a matter related to its environment and previous history.

This approach to the problem has indicated to us that the normal goal of research, instigating the establishment of basic knowledge and of a quantitative point of view must be supplemented by empirical experimentation. The arena of factual data must be supplemented by further related observations, such as of the effect of the addition to carbonyl nickel powder of substances of diverse particulate forms and sizes, and of varying surface-active behaviour. Orienting experiments of this nature are underway with interesting prospects, especially with regard to maintaining a constant shrinkage and decreasing the weight of the plaque in terms of the ultimate ampere-hour capacity per unit volume while maintaining a constant high porosity. It is believed that the indicated direction of the work should not be determined as conclusive and worthy of presentation until the plaques have been impregnated with active mass and tested for capacity and life.

#### (2) Routine Production of Plaques

For the study of the impregnation and electrolysis processes, the assembly of batteries, and the testing of cells, it is desirable to have plaques of reasonably constant properties, including porosity. A large number of plaques were made during the period covered by this report, including the 10.5 sq. in. plaques made in the two piece form with a 3.6

#### 4. Discussion: a. Sintering (Contd.)

by 3.0 inch cavity, 1/8 or 1/4 inch deep, as described in the previous reports. The data for these plaques are summarized in Tables I, II, and VI.

Our previous studies have indicated that there was a possibility of increasing the size of the plate over the earlier projected size without undue detrimental effects upon the life and general behaviour on charging and discharging batteries assembled from the bigger plates. The earlier size was based on the empirical design features incorporated into the design of Nicad and Alcad cells representing design evaluated on the basis of years of experience.

To check the effect of plate height and freeboard in the battery, especially at high charging rates, three plate heights were selected for study. A new three-piece graphite form was designed incorporating top and bottom pieces and a center spacer thereby permitting the future substitution of spacers in various thicknesses for the same top and bottom pieces for various plate thicknesses. The faces of the top and bottom pieces which were in contact with the powder and formed the top and bottom sides of the form cavity were provided with projections for the purpose of forming grooves in the plaque. These grooves, in the vertical direction of the plaque, were incorporated to hold the rod-like separators in place in the assembly of a battery.

With this assembly, the bottom piece and the spacer plate, locked into place by the close fit of the projecting rectangular boss on the bottom piece, formed the cavity into which powder and grid were placed in the usual manner. The cavity is deeper than required and filling is accomplished to the desired level by means of calibrated scrapers. The top piece is placed into position, bringing about a slight compression of the powder at the positions of the vertical projections (forming the grooves on sintering). To eliminate inequalities due to this procedure and especially to eliminate incipient cracking which was noted on sintering, the process of turning each form over after assembly of the compact was adopted. This practice has been found to provide grooves of the desirable sharpness on both sides and to eliminate to a high degree, the tendency for the formation of cracks in the plaques on sintering. In conjunction with this step it was found desirable to level off the powder on the top surface at the theoretical depth of powder provided in the

#### 4. Discussion: a. Sintering (Contd.)

design of the form. With thinner sections, the groove outlines tended to be less sharp, and to broaden out. With thicker sections, there was an increased localised compression which it is believed to lead to cracking of plates and to create unequal stress distribution which could be a factor in plate behaviour in the subsequent treatment and in use.

The plaque sizes have been designated by the sizes FM-1, FM-2, and FM-3 in the order of increasing area, namely 10.7, 12.5, and 14 sq. in. The data are assembled in Tables VII, VIII, IX, and XII.

In connection with making these plates, it was found in the first experiments that the plaque thicknesses, based on the previously established shrinkage in thickness for the various powders and the two-piece 3.6 x 3.0 inch form, were greater than the desired 11/64 inch (4.37mm). The first reaction to this condition was embodied in the fact that the new three-piece forms were heavier than the two-piece forms formerly in use, thereby bringing about a lower final temperature in the same time period than with the two-piece forms. The weights of the two-piece and three-piece forms were 1155 and 1819 grams, respectively. Raising the initial temperature of the furnace improved the end result but the heat capacity of the furnace is insufficient to overcome the increase in weight of the form, and the rate of recovery of the furnace temperature is approximately fixed at a rate which is constant in the high temperature range of the furnace. Due to a delay in obtaining a supply of one inch graphite plates of suitable quality, it was deemed expedient to increase the time of sintering in the furnace.

In making the FM-3 plates, of which the greatest number were made, it was found that after about thirty uses, the spacer showed signs of wear around the upper portion due to the repeated rubbing with the brass scrapers. To overcome this, a steel plate was made to fit over the graphite spacer and this tendency for rapid wear has been overcome. At the same time, the operators became aware of the need to avoid vigorous brushing of the graphite forms in cleaning the form. The wearing of the spacer was accompanied by the formation of cracks at the edges of the plates.



#### 4. Discussion: a. Sintering (Contd.)

##### (3) Effect of time on sintering.

The experience in making the FM size of plaques, described above, led to an investigation of the effect of time within a narrow range. Table V, parts 1 to 4, summarizes the data for the S series, 10.5 sq. in. plaques, in duplicate at sintering times at one minute intervals from 3 to 15 minutes. Table No. XI summarizes the data for the FM-3 series, 14 sq. in. plaques at sintering times from 5 to 15 minutes at one minute intervals.

The shrinkage curves, plotting percent shrinkage as a function of time are represented by two approximately parallel curves with straight sections in the middle portions. Extrapolation to zero sintering time indicates that about one to two minutes are required for sintering the S plaques while at least three minutes are necessary for the FM-3 plaques. The values for the shrinkage of the S plaques at the longer intervals are extremely scattered, indicative of a secondary effect for times longer than ten minutes.

The translation of the data for one size of plate to another will require further physical characterization. The German practice included a hardness determination on a special machine. Checks with the standard Rockwell testing apparatus indicated that plaques sintered for different times could be distinguished but that the dents were made by the adjusting mechanism operating on only 6 Kg spring pressure. Other available machines for hardness testing such as the Baby Brinell are being investigated. An attempt to use the A.S.T.M. penetrometer also failed, probably due to use of a needle as the penetrating medium.

In applying other physical tests which would be suitable for control purposes, consideration must be given to the probable role of the grid in determining characteristic values. This would be true in determinations of tensile strength, etc., as well as possibly in the determination of magnetic properties or electrical resistance. The latter has been favored in our considerations with a view to the development and evaluation of a non-destructive test which would indicate conformation to a standardised product. Such a goal is highly desirable in view of the general reports on the reputed difficulties encountered in German practice with sintered plates. (Reference 7d, 7f, 7g, 7h). The detailed description of their

#### 4. Discussion. a. Sintering (Contd.)

sintering technique never fails to awe one in the possibilities in the evident lack of reproducibility just in the sintering operation. It is believed that the method of piling stacks of compacts with variations in the use of magnesia or graphite plates could hardly lead to reproducible and even sintering. This point is well illustrated by the difference in behaviour shown by compacts of approximately the same size, as shown by the comparison of the tables cited in this section as well as in a comparison of the FM-1 and S plates.

It has appeared to date from available information that the correct degree of sintering is important and that positive plates made from plaques which are too "soft" tend to crumble on use whereas those plates which are too "hard" tend to blister (Ref. 7(d), 7(f), 7(g), 7(h). Occasional plates have been found to show blistering on test and in the few cases in which this has been noted, it was found within the formation cycle or immediately thereafter. Two or three plates, whose behaviour indicated crumbling, were sintered under the same conditions as many others which showed no adverse characteristics and it is believed that the other conditions having to do with impregnating and formation procedure were the responsible factors.

The lack of definition in assessing the qualities of a satisfactory plaque for a given procedure of introducing active mass makes it imperative to evaluate the sintering process in the effect of time. Plaques sintered under this closely similar condition are being saved for qualifying tests and multiplicates are being impregnated and subjected to capacity tests to determine behaviour under standard battery conditions.

#### (4) Perforated Plate Grids.

It was previously reported (Ref. 7 i) that grids of electroformed sheet nickel, perforated or solid, were unsatisfactory, in contrast to the behaviour of perforated rolled sheet, 0.021 inch in thickness. A series of grids with attached lugs having 3/8 inch perforations were cut out of rolled sheet, "A" nickel, of 0.005, 0.010, and 0.015 inch thickness, and of International Nickel Co. electroformed sheet, 0.005 inch in thickness. The results of tests using these grids are summarized

#### 4. Discussion: a. Sintering (Contd.)

in Table No. III. All of the plaques made were satisfactory, and appeared to be as good as could be made by sintering.

It was, therefore, decided to recheck the previous electroformed grids, having the one-half inch perforations. The size of the grid was cut down to allow a clearance of  $3/32$  to  $1/8$  inch around all sides in relation to the walls of the form. These plaques sintered as satisfactorily as those above. The reason for the change from unsatisfactory to good plaques in this case appears to be due to the increased allowance for lateral expansion of the grid during the sintering process. The previously estimated expansion of the grid, based on the known characteristics of massive nickel metal, is clearly an insufficient allowance for grid design. It is probable that restrictions are imposed by the changes occurring in the nickel powder while expansion of the grid takes place during the sintering process. Where the end allowance is insufficient, it appears that the stresses are too great for thin grids and secondary effects are produced resulting in distortion and unsatisfactory plates.

The attention to this characteristic of plaque-making was suspected during work with the wire cloth grids. Success in allowing ample space between the grid and the retaining walls of the form led to the inclusion of this feature in cutting the rolled and electroformed nickel sheet grids. The soundness of this line of thought is amply illustrated by the plaques produced in this series of tests.

The German Durac plates were provided with a grid having a frame retaining a 12 mesh screen of extremely fine wire. It would not be difficult to imagine that the frame of the grid was responsible for the creation of irregularly distributed stresses in the plaque which could be responsible for difficulties in cell behaviour. Electrochemical phenomena are commonly associated with stress phenomena in metals even under ordinary conditions where the metal surface is relatively small and would be accentuated to a high degree with the large area of the order of 0.25 sq. meters per gram exhibited by sintered carbonyl nickel powder.

#### 4. Discussion: a. Sintering (Contd.)

##### (5) Sintered Sheet as Grid.

One of the most surprising features of the work with carbonyl nickel powder is the complete absence of any tendency to sinter to nickel metal in the form of massive nickel metal, electroplated nickel metal, roughened or etched nickel surfaces, or freshly-reduced nickel oxide coatings on metal (even when reduced under "nascent" conditions). This factor was recognized in the German researches (Ref 7 j). It appears that an adhesion of the powder to the grid would improve the strength characteristics, though at this stage of development and testing it is not to be deduced with any degree of persuasion that such an adhesion would present marked advantages.

It occurred to us that if the carbonyl nickel powder could be made to adhere to any object it should be possible to make it adhere to its closest relation, namely a sheet grid made from carbonyl nickel powder. Such orienting tests with solid and perforated sheet, .007 to 0.020 inch thick, made from carbonyl nickel are summarized in Table No. IV. In no case was the tendency for saucerizing characteristic of plaques made without grids entirely absent and in no case was there any appreciable adhesion between the pre-sintered grid and the nickel powder. The sintered powder was readily separated from the grid. For the time being, this project will not be pursued any further.

##### (6) Cracks in Sintered Plaques.

From time to time it has been noted that cracks have appeared in the plaques in an irregular manner, with the possible exception of the condition that with the S series, 10.5 sq. in. plaques, the cracks inevitably appeared in the upper face. These cracks do not appear to interfere with the subsequent treatment of the plaques for the purpose of introducing the active masses, nor in the performance of these plates in cells in those cases where plaques cracked in the impregnation process have been carried through to the testing stage. The principal objection to the cracks, referring mainly to plaques about 4 mm in thickness for the thinner 2 mm plaques seldom show cracks, would seem to be in appearance, a factor which would only show up in dismantling a battery for inspection or overhauling.

#### 4. Discussion: a. Sintering (Contd.)

In the course of our experience, a number of incidents have plainly shown that cracks can be caused or produced by a number of unintended operations. Those having to do with a shaking or jarring of the compact may be caused in handling or transferring the form from the work bench to the furnace, permitting a compact to stand in its form for an extended period probably permitting the formation of incipient cracks by transmitted vibrations, and bumping the form when moving it into the furnace, such as on the roughened casting identification mark found on the hearth of the furnace ledplate. Factors which have been noted to have bearing on crack formation as-a result of operations during the assembly of the compact have to do with the use of improperly flattened grids, movement of the grid after placement especially with the levelling and spreading scrapers, and unequal distribution of pressure on applying the tamping tool.

It has been noted that wear of the graphite form tends to induce cracks and this has been demonstrated conclusively by alternating a worn form with a new form and obtaining an alternating pattern of a perfect plaque and a plaque with a characteristic crack.

In the first tests on the sintering process, when compacts were sintered for up to one hour, it had been noted that there were a larger percentage of plaques with cracks after extended (compared to the 10 minute interval) sintering periods. These have no immediate interest except that it may indicate that recrystallization processes may intensify the enlargement of incipient cracks which may only be detected in plaques made in shorter periods, by microscopic examinations.

#### 4. DISCUSSION

##### b. Impregnation

(1) General Remarks. An extensive volume of information has been amassed on the subject of impregnating porous plaques with the desired active masses of nickel and cadmium compounds. This information is summarized in Tables XIV to XXXIV and is discussed in the following pages. The interpretation of this data is as yet incomplete and the analysis from various points of view is being continued with the idea of confirming the inferences suggested on inspection, of establishing new relationships, of yielding basic data for the engineering factors of design for plant scale production, and of orienting research to the ultimate objectives of optimum battery characteristics and costs. It has been necessary to fill in and extend gaps, as well as to check and confirm experimental results, without awaiting a final analysis, not only to achieve progress and fuller understanding of the underlying processes, but to permit a truer evaluation of the overall picture within the allotment of time and money for this developmental project.

The introduction of active masses into porous plaques made of carbonyl nickel powder has been studied from one aspect on the basis of reproducing the German technique, as described particularly by military representatives (Ref. 7(k)). The multiple cycles of impregnation, cathodic polarisation, washing and drying have resulted in positive and negative plates of somewhat higher capacities than reported for the Durac plates, namely 0.133 A.H. per sq. in. per mm (Ref. 7(l)). Only one plate has been studied with a view to determining maximum possible capacity, which appears to be higher than supposed. In this phase of the investigation, it is a problem of balancing the specifications desired for different battery services against cost and the time required to evaluate stability in service.

Sufficient data has been collected, and a sufficient number of plates have been made and tested to warrant the statement that alkaline batteries with sintered positive and negative plates could be produced in quantity in a suitably designed plant. This statement would be incomplete if the opinion, based on our experimental results, were not expressed that the process of impregnation can be improved and simplified, especially with the ultimate prospect of decreasing the expected manufacturing cost. This

#### 4. Discussion: b. Impregnation (Contd.)

view has led to further examinations of the fundamental chemical processes involved in the impregnation procedure.

In deviating from the established technique of vacuum impregnations with multiple cycles to other impregnation techniques, such as at atmospheric pressure, there is encountered a sphere of undefined standards of comparison. The vacuum technique had been adopted in German practice to take advantage of the usual high efficiency of vacuum impregnation techniques in general (Ref. 7 (m)). Offhand, there is no apparent reason why the life of a plate impregnated at atmospheric pressure should differ from that of one prepared by a vacuum treatment, yet experience with all types of alkaline batteries indicates a background of evidence of marked variations in properties due to particular processes and there is hence a very strong reluctance to use new procedures without prior and thorough testing. In this case it is clear that only life cycles of testing of plates in the form of assembled batteries can furnish the ultimate answer. With such a time factor to consider, it is evident that this research program must of necessity represent a compromise in its extension and its intensive development.

##### (2) Impregnation of Negative Plates at Atmospheric Pressure.

The impregnation of plaques with cadmium nitrate solution and their subsequent electrolytic polarization to precipitate in their pores the water-insoluble cadmium hydroxide may properly be discussed here and apart from the corresponding procedure used for the positive plate impregnation. In the treatment of porous nickel plaques with cadmium nitrate, nickel tends to dissolve in the cadmium nitrate solution as evident for instance by the color change of the solution during the impregnation of the plaque. It has also been found that a solvent consisting of ammonium hydroxide and ammonium chloride in water dissolves active mass out of the impregnated plates. The negative active mass yields soluble nickel whereas treatment of sintered nickel plaques (without active mass) does not show soluble nickel (except under particular conditions of concentration and temperature). The precipitated active mass will, therefore, consist of cadmium hydroxide with smaller and varying amounts of nickel hydroxide. This condition introduces a secondary variable, namely specific ion effects, which may change the reaction mechanism for the two types of plates.

#### 4. Discussion: b. Impregnation (Contd.)

In the present study, the principal variables introduced have been plaque thickness, time, temperature and the cycles of treatment. Previous studies (Ref 7n) indicated a marked gain in weight with increased time of impregnation at room temperature. These results have been extended for 2 and 4 mm. thick plates in experiments summarised in Tables XIV, XV, and XVI. The gains in weight of the plates may be roughly divided into two groups, namely for impregnation periods up to one hour and for periods longer than one hour. Within each group there are however irregular increases and decreases in the gains.

This feature of irregularity, which is also characteristic of the positive plates impregnated with nickel nitrate solution, seems to be explained by a phenomenon observed in a special study. Plaques were permitted to absorb solution by capillary action, in such a manner that each plaque was above the solution and the action could be observed. There appeared to be a recurrent cycle in the evolution of gas bubbles. The interaction of the plaque and the nitrate solution produces gas which collects as bubbles within the pores and which are not discharged till sufficient pressure has been developed to overcome the retaining capillary forces. The solution will obviously be displaced by the growing gas bubbles and hence the plate will be loaded alternately to different degrees corresponding to the accumulation of gas. Thus the amount of active material which can be precipitated in the pores by the subsequent polarization process will depend on the particular extent of the gas accumulation at the moment of removing the plate from the solution.

No consideration has yet been given to the effect of a variation in the weight of the negative active material due to changes in the ratio of cadmium to nickel in the precipitated hydroxides. This can only be determined by analysis, a difficult problem since it is required to differentiate between nickel metal and nickel hydroxide. Such analyses are underway, as it is desired to determine the coefficient of utilisation of the cadmium in the negative plates on the basis of analytically-determined values. The error in assuming that the gain in weight is cadmium hydroxide only, is probably small, since the gain in weight due to conversion of nickel to hydroxide is balanced (and perhaps exceeded) by the loss of nickel by the plaque to the impregnating solution,



#### 4. Discussion: b. Impregnation (Contd.)

For convenience the results have been summarized in Table No. 4. A single impregnation at atmospheric pressure and room temperature results in the introduction of about as much active mass for impregnation periods up to one hour as in a single vacuum impregnation. This amount of cadmium hydroxide is of the same order of magnitude as calculated from the volume of cadmium nitrate solution equal to the pore volume of the plaque.

The increase in the amount of active mass introduced by the impregnation in periods longer than one hour indicates an increase in the cadmium concentration in the pores, either by the formation of soluble "basic" cadmium nitrates or by the precipitation of insoluble basic cadmium nitrate or hydroxide. In either case there can be an equalization of the cadmium nitrate concentration by diffusion into the pores of the plate from the ambient solution. The increase in weight beyond the one hour period amounts to about  $\frac{2}{3}$  of the total gain for a double vacuum impregnation of a 4 mm. plate and at least 0.8 to 0.9 for a 2 mm. plate. This difference between the two plates probably has a distinct meaning in terms of the mechanism of the process. The data permits the conclusion that in the case of a single impregnation of a plaque with cadmium nitrate solution to produce negative plates, an immersion period of 1 hour is sufficient and no appreciable gain seems to be made in the extension of the period to as long as 24 hours.

In the case of positive plates, elevation of temperature seemed to be of some benefit, and it was decided to test this feature for the negative plate impregnation. The data, summarized in Table No. XXIII for immersion periods of 1 to 6 hours shows a definite decrease in the gain in weight, averaging about 3 to 4 grams per plate for the single impregnation. There does not appear to be any advantage in raising the temperature for this process and there was no change in the physical appearance of the plates.

To check the behaviour on multiple impregnations, the plates were recycled for a total of two or three impregnations, as summarized in Part 2 of Tables XIV and XV, but restricting the longest time to 1 hour except for the 24 hr. plates. The plates which had been impregnated for shorter periods of time were impregnated in the subsequent repeat cycles for the same time as originally. For convenience of reference, the weight gains are summarized in Table No. 5.

4. Discussion: b. Impregnation (Contd.)

Table No. 4

Single Atmospheric Impregnation of Negative Plates  
Gain in Weight as a Function of Time at Room Temperature

Time	Total Gain in Weight, g ( $\text{Cd}(\text{OH})_2$ )		
	Table XIV 2 mm. plates	Table XV 4 mm. plates	Table XVI 4 mm. plates
Hrs - Min			
5	6.17	14.08	
10	5.65	12.13	
15	5.89	14.75	
30	7.01	15.96	
1	9.64	19.81	19.20
2	9.39	18.66	19.67
3	9.78	18.74	19.22
4			17.98
5			17.31
6	10.72	19.25	19.02
24	10.44	17.76	
48	8.44	18.72	

For comparative purposes, the following gains on impregnation are summarized for plaques of the same series as above (Table XXVI)

No. of Impregnations	Total Gain in Weight (g)	
	2 mm. Plates	4 mm. Plates
1	5.75	14.31
2	11.32	27.29
3	15.68	37.57
4	19.70	46.42

4. Discussion: b. Impregnation (Contd.)

Table No. 5

Multiple Atmospheric Impregnation of Negative Plates

Gain in Weight as a Function of Time

See Tables XIV & XV, Part 2

Time Hrs. - Min.	Total Gain in Weight, g. (Cd (OH) <sub>2</sub> )			
	2 mm Plates		4 mm Plates	
	2 cycles	3 cycles	2 cycles	3 cycles
5	11.67	17.11	24.10	32.98
10	11.79	16.59	22.80	31.70
15	11.31	15.41	25.39	34.26
30	11.15	17.02	24.71	34.88
1*	12.57	nd	24.14	nd
24	13.65	nd	27.83	nd

\*Average of 4 plates

#### 4. Discussion: b. Impregnation (Contd.)

The previous difference in grouping at the one hour line appears to hold for the second impregnation for which comparative data are available. There is, however, a marked difference in behaviour on the second and third impregnations as the gain in weight shows a higher rate of decrease than is evident for multiple vacuum impregnations. For the 2 mm. plates, the initial higher gain in the first impregnation permits an equalisation with vacuum impregnation, cycle for cycle, whereas for the 4 mm. plates there is a definite lag in weight gain in comparison with the results of a comparable number of vacuum impregnating cycles. This is not a particular disadvantage so long as multiple cycling is required to attain capacity since the gain in ampere-hour capacity on impregnation with active mass appears to be greater for the negative plates than for the positive. The ultimate need will be determined by the relative values of the coefficients of utilisation and of any desired excess in negative plate capacity over that of the positive.

#### (3) Impregnation of Positive Plates at Atmospheric Pressure. Effect of Immersion Time.

Systematic results on the effect of immersion time have previously been presented (Ref. 7(o)) for 2 and 4 mm. plates at elevated temperatures for nickel nitrate solution of usual concentration and for one containing 95% by weight of nickel nitrate hexahydrate. The latter was found to be very close to yielding the desired capacity in a single impregnation of the 4 mm. plates but it appeared that extended periods, of say 6 hours, caused the 2 mm. plates to buckle while immersed in the nickel nitrate solution. It was desired to extend the experiments to room temperature to obtain an overall picture of the process.

For immersion periods of 1 to 6 hours, the gain in weight shows a small irregular increase with time, and with variations in accordance with the discussion above, under the previous heading. The total increase in weight on one impregnation approaches the value obtained on two cycles of impregnation in a vacuum, thereby exceeding by an appreciable amount the calculated gain in weight, 9.5 g  $\text{Ni(OH)}_2$ , which can be derived from a volume of nickel nitrate solution equivalent to the plaque pore volume. The data, summarized in detail in Table No. XX, have been abstracted to show the final results in Table No. 6.

4. Discussion: b. Impregnation (Contd.)

Table No. 6

Atmospheric Impregnation of Positive Plates  
Effect of Time from 1 to 6 hrs. at Room Temperature.

4 mm. plates

Time hrs.	Gain in Weight g $(\text{Ni}(\text{OH})_2)$
1	13.40
2	13.69
3	14.68
4	13.07
5	14.83
6	15.38

Gain in weight of like plates by vacuum impregnation.

No. of Impregnations	Gain in Wt. g $(\text{Ni}(\text{OH})_2)$
1	8.46
2	15.98
3	22.84
4	27.25

#### 4. Discussion: b. Impregnation(Contd.)

The effects of extending the impregnation periods have also been studied in view of our early orienting experiments in which it was noted that the coefficient of utilisation could be interpreted to mean that the active mass precipitated consisted of nickelous oxide instead of nickelous hydroxide. The first set of experiments with plaques belonging to the same series as used above resulted in an irregular series with small gains for 24 hrs., and only approaching the results for the 1 to 6 hr. period in 2 or 5 day periods. These results showed a greater irregularity than in previous tests, which may have been due to lower temperatures for the period of testing, (especially when standing overnight is included). The experiments, summarized in Table XVIII, were repeated using plaques made from the latest powder. These plaques, for which the experimental data are summarized in Table No. XXXI, showed a gain in weight in 24 and 48 hrs. equivalent to two cycles of vacuum impregnation, or slightly higher than the previous results up to 6 hours of immersion. This gain in weight is also of about twice the magnitude to be expected from the nickel content of a volume of solution equal to the pore volume of the plaques.

#### (4) Impregnation of Positive Plates at Atmospheric Pressure. Multiple Impregnations.

As a result of the studies with the negative plates, it was decided to carry out multiple impregnations of positive plates for one hour periods. The experimental data with 2 and 4 mm. thick plates receiving up to four impregnations are summarized in Tables No. XX and XXI. The results on the gain in weight of the plaques are shown in Table No. 7, including results of vacuum impregnations of comparable plaques. The data indicate that the multiple cycle vacuum type impregnation succeeds in introducing a greater amount of active mass into the plaques but this added amount can not be considered appreciable and could easily be equivalent, depending on the exact value of the coefficient of utilization found when tested electrically for capacity. It is of interest to note again that impregnation after the first cycle favors the vacuum process, indicating that wetting characteristics of the plate are changed by the introduction of active mass.

4. Discussion: b. Impregnation (Contd.)

Table No. 7

--Multiple Atmospheric Impregnation of Positive Plates

No. of Impregnation Cycles	Gain in Weight, g $\text{Ni(OH)}_2$			
	2 mm. Plates		4 mm. Plates	
	Atmos.	Vacuum	Atmos.	Vacuum
1	4.12	3.36	8.93	8.46
2	7.03	6.66	15.13	15.98
3	9.97	9.82	21.60	22.84
4	12.28 *	12.77	26.52	27.25

\* Average of three.

#### 4. Discussion: b. Impregnation (Contd.)

The experiments described above appear to be highly favorable to the possibilities of the use of atmospheric impregnation.

##### (5) Impregnation of Positive Plates at Atmospheric Pressure. Effect of Temperature.

Further data was obtained by impregnating plaques at 120° F as shown in detail in Table No. XXII. There is a decrease in the weight gains in comparison to the results at room temperature. The values are compared in Table No. 8 with the values for gain in weight on impregnation obtained at other temperatures. No advantage appears to be gained unless test results show an increase in the coefficient of utilization with increased impregnation temperature. Such an increase, even at the previously determined figures would not counterbalance the lower values of the gain in weight on impregnation at elevated temperatures.

##### (6) Impregnation of Positive Plates at Atmospheric Pressure and Constant Acidity.

To check the possibility that impregnation over an extended period of time and at atmospheric pressure - of long duration compared to the five minute vacuum impregnation - was being affected in some way by the acidity of the solution, two sets of five plaques in each were impregnated, the acidity being kept approximately constant. This was done by the regulated addition of a strong nickel nitrate-nitric acid solution. The experimental data are summarized in Table No. 19. These plates actually showed a lower weight gain over comparable plates in experiments in which the acidity was permitted to change during the impregnation by limiting the control to a single initial adjustment of the acidity. The gain in weight showed an average value, under conditions of controlled acidity, of about 25% less than under conditions of no control of acidity change during impregnation.

##### (7) Vacuum Impregnation-Routine Plate Production.

A total of 165 positive plates and 30 negative plates have been impregnated by the multiple vacuum procedure and for the purpose of providing plates for test cells and batteries. The experimental data, in



4. Discussion: b. Impregnation (Contd.)

Table No. 8

Atmospheric Impregnation of Positive Plates  
Comparison at Various Temperatures  
4 mm plates

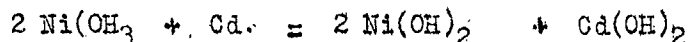
Duration of Impregnation hrs.	Gain in Weight, g		
	68°F Table No. XVII	120°F Table No. XXII	210°F Ref 7(o) Table No. XX
1	13.40	11.80	10.74
2	13.69	12.69	11.98
3	14.68	12.32	13.46 *
4	13.07	11.71	nd
5	14.83	10.77	nd
6	15.38	10.95	12.98 *

Note (\*) These values may be the result of an increase in concentration of the nickel nitrate solution due to evaporation.

#### 4. Discussion: b. Impregnation (Contd.)

summary form, are given in Tables XXIV, XXV, XXVI, and XXVIII. The data available will permit an exhaustive analysis of the vacuum impregnation process. The necessary calculations however are incomplete at this time so that their presentation and complete discussion is postponed. The delay will permit a correlation with the results of tests to determine electrical characteristics.

The higher rates of impregnation for the negative plates in comparison with the positive plates is illustrated by the results. The gains in weight on impregnation have been converted to theoretical capacities in ampere-hours on the basis of the electrochemical conversion of energy for the reaction:



For a like number of vacuum impregnations, with all conditions constant except for the solutions, which are the source of the active masses, the accumulation of active mass by the negative plates is at about twice the rate of accumulation of active mass by the positive plate. This is immediately evident from the data summarized in Table No. 9.

#### (8) Vacuum Impregnation. Effect of Sintering Time.

It has been indicated that the sintering time in preparing the plaques is of vital importance in the ultimate behaviour of a plate, particularly the positive plate, in the alkaline battery. This subject has been treated in the appropriate prior section of the Discussion. The fundamental importance of evaluating this factor from the standpoint of battery life can not be overestimated and this series of experiments is a start in its proper characterization.

Compacts of the FM-3 size were sintered for periods of from 5 to 15 minutes; the data for the plaques have been summarized in Table XI. These plaques were impregnated by the vacuum procedure, keeping all conditions constant. The experimental data have been summarized in Table No. XXVII.

Examination of the results shows a decrease in the weight gain which is regular from the plaque sintered in the shortest time of

4. Discussion: b. Impregnation (Contd.)

Table No. 9

Vacuum Impregnation, Comparison  
of Positive and Negative Plates

Plate Description	No. of Impregnations	Table No.	Theoretical Capacity-AH	Theor. Capacity AH/sq.in/mm.
Neg, FM-3	4	XXIV	21.59	0.339
Pos "	4	XXV	10.88	0.171
Neg, S, 2 mm.	3	XXVI	5.80	-
Pos, S, 2 "	3	"	2.75	-
Neg, S, 2 "	4	"	7.08	-
Pos, S, 2 "	4	"	3.76	-
Neg, S, 4 "	3	"	14.15	-
Pos, S, 4 "	3	"	6.40	-
Neg, S, 4 "	4	"	16.56	-
Pos, S, 4 "	4	"	8.09	-

#### 4. Discussion: b. Impregnation (Contd.)

5 minutes to the plaque sintered in 15 minutes. This decrease corresponds to a change in theoretical capacity from 16.0 A.H. to 12.6 A.H. However, the plaques show a regular decrease in thickness which follows the same course. The results have been calculated in terms of the unit weight gains. Table XXVII shows the results in terms of grams gain per cubic centimeter of pore volume. The average value is 1.27 g per cc with a maximum deviation of 0.05. The time of sintering has, therefore, not wrought any changes in the plaques which have an effect upon the impregnation process.

If it is assumed that the density of nickelous hydroxide can be taken at the value of the precipitated forms showing distinct diffraction patterns, namely 4.10 (Ref 7 p), the volume of the nickelous hydroxide is 0.31 cc per cc of pore volume, so that 31% of the available pore volume is filled on four impregnations.

All of the plates of this series of tests have been made up into test cells and their performance over an extended period will be observed.

#### (9) Vacuum Impregnation of Porous Graphite Plates.

An understanding of the impregnation process will depend to a large extent on the isolation and measurement of the effect of individual independent variables. The elimination of the reaction between the nickel metal of the plaque and the solution seemed to be a desirable starting point, particularly in view of the recognition of the function of the evolved gas in changing the degree of impregnation with time. This line of thinking led to the possibilities of porous graphite plates, which are available commercially. It is recognized, however, that graphite is not completely inactive in electrochemical reactions, especially in its tendency to react with oxygen to form graphitic acids, often in the form of surface compounds.

Porous graphite plates are available in various grades, having an approximately constant effective porosity, but varying in pore diameter. The plates used in this series of tests varied in pore diameter from 33 to 140 microns and had an effective porosity of 48%. These sizes fall into the classification "macroporous", that is with the bulk of their pores with diameters greater than 5 microns (Ref. 7 q). The variation

#### 4. Discussion: (b) Impregnation (Contd.)

in pore diameter is of direct interest, especially with regard to establishing whether or not it would be desirable to expend effort in learning how to control this factor if there proved to be an optimum distribution for maximum plate capacity.

The experiments were carried out in the usual manner, impregnating for four cycles with nickel nitrate solution. The experimental data are shown in Table No. XXX. The results show differences in behaviour, not only in the first cycle of the impregnation, but in the overall picture. The grade 60 plaque with a pore diameter of 33 microns showed a steady and marked decrease in weight gain on successive impregnations, whereas all of the other grades showed a variation similar to that noted with carbonyl nickel plaques, particularly in their tendency to show a substantial weight gain on the third cycle when the weight gains on the first and second cycles were comparatively low.

The overall gain expressed in terms of the percent of the pore volume occupied by the nickelous hydroxide varied from 18 to 27%, whereas comparable results for nickel plates showed 31%. This may be the result of the difference in wetting and penetration by the solution or to the absence of the reaction occurring in nickel plaques between the sintered metal and the solution. This line of experimentation will be continued and extended, especially to the negative plate so that the possible effect of nickel in the impregnation process will be eliminated. These carbon plates will also be tested as cells for capacity and electrochemical behaviour.

#### (10) Cathodic Polarisation in Lithium Hydroxide.

Inasmuch as it is known that relatively small amounts of lithium ion, added to the electrolyte of alkaline cells, may have beneficial effects on their capacity and life and since the mode of its action has not been clearly defined, preliminary thought suggested the possibility that this beneficial effect could be incorporated by polarization of the plates in lithium hydroxide solutions. Accordingly, a series of plaques was subjected to four impregnation cycles, during which each polarization was carried out in lithium hydroxide solution. The experimental data are shown in Table No. XXIX. All of the plates have been

#### 4. Discussion: b. Impregnation (Contd.)

made up into test cells. The weight gains are of the same order as found in the usual procedure with polarization in potassium hydroxide solution, thereby eliminating the possibility that the active masses tend to absorb or combine with appreciable amounts of lithium ion.

It was noted that the fumes produced in the lithium hydroxide polarizations are strong irritants to the nose and throat so that suitable precautions should be applied in using this reagent.

#### (11) Pore Volumes from Drying Data.

For the proper design of a drying unit, the amount of water to be removed from the plates following the washing step must be known. In the course of determining the water content of typical plates for which the data are shown in Tables No. XXXII and XXXIII, the figures were converted to volumes and pore volumes calculated by adding to this value the volume of the precipitated active mass. The pore volumes found in this way are smaller than those calculated from the measurements of the plaques, but are within 10 percent of these values. This could mean that the actual densities of the precipitated hydroxides are lower than used, basing the values on crystalline forms, but in any event the data show little loss in available pore volume and it can be expected that the free space in the impregnated plates will be filled with electrolyte in the cells. This approach should be used in any detailed analysis of the role of diffusion in the behaviour of the alkaline sintered plate battery.

#### 4. DISCUSSION:

##### c. Testing

(1) General Remarks. The testing procedure which had been adopted for determining the electrical capacity of plates was described briefly in a previous report (Ref. 7r). Its aim was to set up means for evaluating plate capacities in terms of the theoretical capacities calculated from weight gains, which is equivalent to the determination of the coefficients of utilization. The design of the test cells was different in some respects from the usual alkaline batteries. The volume of electrolyte provided was greater and the free space in the cans permitted an electrolyte circulation during charging and discharging cycles. There was an absence of tension on the plate group except for the rubberband or string with which the plate group was tied, and the plate separation was not held rigidly to the standard value of 0.03 inch generally used in alkaline batteries with the pocket type of plate. The testing procedure did not conform to rigorous schedules of charging and discharge cycles and rates. This state of affairs, for which no apology is intended, was in part ascribable to the large number of plates of differing capacities which were subjected to electrical testing, and in part due to the pioneering aspect of the work. There is a gap in background on which to extrapolate individual plate capacities to ultimate performance in batteries, and it would be false economy in planning to expect like performance from the sintered plate in comparison to the pocket plate merely on the basis of the expected higher coefficients of utilization of the active masses of the sintered plates. It also appears that testing should be predicated to a certain extent on the ultimate specifications for which a battery is to be used. It is desirable to establish the relationship between plate testing results and battery design.

The most bothersome feature of the testing program has been in the irregularity of the capacities found for an individual plate over a consecutive number of charging and discharging cycles. This feature is observed apparently whenever a detailed analysis is made of capacity tests of alkaline batteries, whether of the sintered plate type or of the pocket type and has been noted in the work of other laboratories.

#### 4. Discussion: c. Testing (Conti.)

The selection of an absolute value for the plate capacity becomes an arbitrary procedure, within certain limits, under such conditions. In any logical approach to this subject, it is necessary to scrutinize the testing procedure in all its phases and conditions.

A typical example of the possible effect of a testing condition is illustrated by the introduction of the variable, the interval of time that the battery is on open circuit from the end of charging to the start of discharging. The open circuit potential of an alkaline battery falls on standing immediately after discharge, and although the capacity loss due to this change is small, its value is within the range of capacity variation noted. The choice of a period of standing during testing will depend on the use to which a battery will be applied. For Diesel starting, the conditions would call for only a few minutes of standing on open circuit; for a miner's lamp, the choice would be dictated by the practical considerations of the charging schedule and delivery of the lamp to the user.

A different point of view makes it conceivable that the variation in capacity on repeated cycling is a function of the plate structure. Considering the active mass of a positive plate, charging changes nickelous hydroxide to nickelic hydroxide while discharging reverses the reaction. Little is known about the nature of the reaction from the point of view of the behavior and changes of the individual hydroxide particles. The apparent reversibility of the reaction may well hide changes in the sizes and aggregation of individual hydroxide particles. If the reversibility were perfect, it might be expected that the coefficient of utilisation would always be near or at 100%. The lower values, especially with multiple impregnations of sintered plates, may well permit a condition whereby the cycle of charging and discharging opens and closes new pores thereby changing the accessibility to electrochemical reaction. An insight into these processes may provide the cue for new approaches to the general problem of making improvements in batteries.



#### 4. Discussion: c. Testing (Contd.)

The suggested changes in testing procedure are being applied in the program of determining plate capacities. It is not proposed to discuss these changes prior to a more thorough study of the results. There are still a number of arbitrary features, which, however, it is suspected will be an ever-present source of provocation for further excursions into the nature of the fundamental laws governing secondary batteries.

(2) Pocket Type Cells. Various combinations of pocket type cells were made up, particularly with the types of pocket plates which were used in combination with sintered plates for testing their capacity. The results are shown in summary form in Table No. XXXV. These cells showed the typical variation in capacity on consecutive cycles of charging and discharging. The average capacity of the cells exceeded the rated capacity of the plate group with the lower rating in all tests. Attention is called to the capacities well over the rated figures when the negative plate is provided with a positive plate-group with ample excess capacity. The combination of a negative plate with a positive plate group of twice the rated capacity gave capacities of 150% of the rated capacity of the negative plate.

Tests in which various anions, such as sulfate, chloride, and carbonate were added to the electrolyte did not show any specific effects over 22 cycles of charging and discharging. The same feature had been noted for the nitrate ion, as reported in the Quarterly Progress Report, 30 Sept. 1947. Table XXVI, page 53. This ion would be expected as a possible contaminant of sintered plate cells made up by impregnating with nitrate solutions. After 20 cycles, it was found by analyses of the electrolyte that the nitrate had been eliminated from the electrolyte. These tests indicate that up to the limits of concentration used in the cells, foreign anions will have little effect on the capacities determined for the sintered plates. An examination of the capacity data for the cells with nitrate added to the electrolyte showed a diminishing loss of capacity on standing on open circuit (usually over weekends). This would indicate that nitrate ion will react with charged plates, thereby reducing capacity and in this case, providing a means for its elimination from the electrolyte.

#### 4. Discussion: c. Testing (Contd.)

(3) Effect of Sintering Time on the Capacity of a Plate. It was shown, in Table XVIII of the 30 Sept. 1947 Quarterly Progress Report that sintering times longer than 15 minutes caused a decrease in the theoretical capacity attained in three vacuum impregnations. The average capacities show a steady decrease as a function of sintering time; the testing data are summarized in Table XXXIX. Up to a 20 minute sintering period, the coefficient of utilization is 85%, dropping to 81% for the 25 and 30 minute sintering period. The latter periods correspond to sintering times for which the shrinkage on sintering has reached a steady value.

It is now recognized that sintering periods beyond 15 minutes will not be of particular importance. However, the plates sintered up to 30 minutes have not shown any tendency toward disintegration or particularly, of blistering, in variance with the suggestions that "hard" plates produced with long sintering periods and by high temperatures are characterized by such defective behaviour. These experiments would indicate other causes. On the other hand, the total number of impregnations was limited to three cycles, which may suggest that there is a limiting value of the pore volume which may be occupied by the active mass. It is possible that a heavier loading of these plates, such as by a fourth impregnation cycle, might bring about the reported blistering effect on utilization of the plates in batteries.

(4) Positive Plates Impregnated at Atmospheric Pressure.  
Single Impregnations. Selected plates from the groups impregnated at 99°C in 50 and 60% nickel nitrate ( $\text{Ni}(\text{NO}_3)_2$ ) solution were assembled in test cells with pocket type negative plates. The data are shown in summary form in Tables No. XXXX and XXXXI. The testing results show that positive plates may be impregnated in strong nickel nitrate solution at 99°C in a single treatment at atmospheric pressure to yield capacities approaching the values indicated for the performance of the Durac plates. Considering the orienting nature of these experiments, it would be desirable to carry out further tests, though the process will apparently be unsuitable for thin plates owing to the tendency of the 2 mm plates to buckle during immersion for periods longer than three hours. The coefficients of utilization had about an average value of 82%, indicating again that the relationship here is dependent upon the percentage

#### 4. Discussion: c. Testing (Contd.)

occupancy of the pore volume by the active mass. This point has been emphasized repeatedly, as there is a possibility that calculations based on active mass to electrolyte proportions using the usual electrochemical expression of the reactions may put a meaning into the phenomenon.

A number of the plates were transferred to test cells having increased negative plate capacity, with little change in the capacities found. This procedure, along with the information on testing pocket type negative plates, eliminated the possibility that insufficient negative capacity was used in this particular series of tests.

(5) Negative Plates Impregnated at Atmospheric Pressure. Data on the testing of negative plates impregnated in 2 and 3 cycles at atmospheric pressure are shown in Table No. XXXXIII for both 2 mm. and 4 mm. plates. In order to provide sufficient positive plate capacity in most of these tests, it has been necessary to use groups of four positive pocket-type plates, so that each sintered negative plate was surrounded on two sides by two pocket-plates, each of the same area. The 2 mm. thick negative plates showed a coefficient of utilization of 92% for two impregnations, while a third impregnation dropped the value for the coefficient to 84%. This appears to be an effect similar to that found in the positive plates on multiple impregnation or on attainment of capacities approaching the figure for the Durac plates. The capacity of the negative plates with three atmospheric impregnations has reached a value of 0.24 A.H. per sq. in. per mm., exceeding the corresponding value of the positive plates and confirming the higher rate of impregnation of the negative plates as shown by the weight gains or theoretical capacities. This feature is the reverse of what was expected on the basis of the descriptions of the manufacture of Durac negative plates (Ref. 7K). It was stated that the negative plates sometimes received five impregnations to insure the proper weight gain. It is conceivable that such a condition might have been due to the manufacturing procedure involving delays in transferring the plaques from the impregnating solution to the cathodic polarization process.

(6) Multiple Impregnation of Positive Plates at Atmospheric Pressure. Table No. XXXXII summarizes data on cells with positive plates which had received four cycles of impregnation at room

#### 4. Discussion: c. Testing (Contd.)

temperature and atmospheric pressure, and compared with control tests on plaques from the same batch impregnated by the vacuum procedure. The tests show that the atmospheric impregnation for four cycles, with plaques of 2 and 4 mm. thickness, results in capacities at the same coefficient of utilization as for vacuum-impregnated plates. The actual capacities for the plates impregnated at atmospheric pressure are slightly lower than the capacities of the vacuum-impregnated plates, in accordance with the indications from the theoretical capacities.

The results to date indicate that the theoretical capacities, based on weight gains on impregnation of plates, can be used to predict ultimate capacities without going through the tedious testing procedure. This would permit the concentration of the test work on multiplate batteries for which the other important factors, such as life, plate durability, charging characteristics, discharge at various rates, shelf life, etc., can be adequately and more thoroughly evaluated.

(7) Miscellaneous Tests. Tables XXXVI to XXXVIII show data for various plates, for which the results have not yet been fully co-ordinated. The results of Table XXXVI refer to FM-3 positive plates which have been charged and discharged at various rates, which will be co-ordinated with the results of tests on similar plates assembled in multiple plate cells and twin batteries. These data are included to show the various phases of work underway and to indicate that answers to some of the questions posed by testing procedure will be forthcoming in the not too distant future.

#### 5. Conclusions and Recommendations.

The Summary, section 2, gives a brief statement on the conclusions indicated by the work performed in this quarter. It is proposed that final conclusions should await the rounding out of the experiments to fill the apparent gaps of information. Two very broad avenues of research and development are indicated by the work to date, namely investigations into the fundamental nature of the electrode processes of the nickel-cadmium alkaline battery in relation to the performance of sintered

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5. Conclusions and Recommendations (Contd).

→ plates, and the extension of the processes involved in producing sintered plates on a large scale with a view to establishing low-cost quantity production. It is proposed that the work continue along these lines and the completion of those experiments suggested by the experimental work presented in this and previous reports. ↲

6. Acknowledgments

The development program is subdivided in accordance with the subject headings under DISCUSSION, namely sintering, impregnating, and testing. The experimental work in each of these fields has been tabulated by Messrs. L. Fay, R. Hodgkinson, and W. Ryan, respectively. Correlation of impregnation data is to a large extent the work of Mr. Hodgkinson and he shares responsibility for the ideas expressed on that subject.

7. References

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- c. Quarterly Progress Report, 30 Sept. 1947.
- d. G. B. Ellis. German Battery Industry. PB13806.
- e. G. Grube and H. Schlecht. Sintering of Metal Powders. Z. Electrochem 44, 367-374 (1938).
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- g. L. E. Lighton, Aircraft Batteries. PB22928, page 22, page 25.

7. References (Contd.)

h. F. X. Brennan, Investigation of Foreign Electrical Batteries. Project No. DQ126. Oct 1947, page 2.

i. Quarterly Progress Report, 30 Sept. 1947, p. 9.

j. Quarterly Progress Report, 30 Sept. 1947, p. 11.

k. See 7d, f, g. h. Also P. E. Flehm, Nickel Cadmium Storage Batteries in Germany, PB42771 (1946).

l. Annual Progress Report, 30 June 1947, p. 50.

m. Statement by Dr. A. Boars, conference 14 Nov. 1947, Squier Laboratory, Fort Monmouth.

n. Quarterly Progress Report, 30 Sept. 1947, pp. 21-2, 50.

o. Quarterly Progress Report, 30 Sept. 1947, Tables XIX and XX, pp. 46 and 47.

p. R. W. Cairns and E. Ott. X-ray Studies of the System: Nickel-Oxygen-Water. I. Nickelous Oxide and Hydrate. J. Am Chem. Soc. 55, 527-33 (1933); CA27, 1795.

q. R. B. Anderson, et al. Kieselguhrs. Suitability as Carriers in Catalysis. Ind. Eng. Chem 39, 1627 (1947).

r. Quarterly Progress Report, 30 Sept. 1947, pp. 23-24.

  
Research Director

# 8. Appendix

Table I

## Sintering; Routine Production for Impregnation Tests

### 10.5 SQ IN BY "2" mm Plaques

Experiment No.	S	383-414	Average	High	Low
Date	1947	9/12-9/15	- - - - -	- - - - -	- - - - -
Record	Vol.V	94-101	- - - - -	- - - - -	- - - - -
Operator		Paul Stahlberg			
No. of Plates Prepared		20	20		
<u>Compact Preparation</u>					
Powder, Type		P-21	- - - - -	- - - - -	- - - - -
Weight Sintered	g	- - - - -	14.4	15.7	12.9
Apparent Density	g/cc	- - - - -	0.70	0.77	0.63
Grid, Type		8x8 Nickel Wire, Welded Lug, 0.028" dia Wire, Annealed			
Weight	g	- - - - -	15.7	16.2	15.1
Form		3.60"x3.00" x 0.125"	- - - - -	- - - - -	- - - - -
<u>Experimental Conditions</u>					
Sinter Temp, Initial	°F	1700°	- - - - -	- - - - -	- - - - -
" " Average	°F	- - - - -	1661	1678	1645
Sintering Time	min.	10-	- - - - -	- - - - -	- - - - -
<u>Results</u>					
Thickness	mm	- - - - -	2.22	2.66	1.93
Shrinkage in Thick.	%	- - - - -	30.5	39.2	26.6
Plate Area	sq.in.	10.51-	- - - - -	- - - - -	- - - - -
<u>Calculated Results</u>					
Plate Volume	sq.in.-mm	- - - - -	23.33	27.96	20.29
Pore "	cc	- - - - -	11.70	14.63	9.57
% of Apparent Sinter Volume		- - - - -	87.4	89.6	84.4
% of Final Plate Volume		- - - - -	77.5	81.2	73.2
Net Pore Volume, cc/sq.in.-mm		- - - - -	0.500	0.523	0.472
Unit Plate Weight, g/sq.in.-mm		- - - - -	1.237	-	-
" Powder "	"	- - - - -	0.615	-	-
" Grid "	"	- - - - -	0.672	-	-

See Table XIII, 16 for Notes

8. Appendix (Contd.)

Table No. II

## Sintering; Routine Production for Impregnation Tests

10.5 SQ IN BY "4" mm plates

Experiment No.	S	387-416			417-442		
		Average	High	Low	Average	High	Low
Date	1947	9/12-9/16			9/16-9/17		
Operator		P.S.			P.S.		
No. of Plates Prepared		14			26		
<u>Compact Preparation</u>							
Powder, Type		P21			P21-1		
Weight Sintered	g	30.7	31.7	29.2	33.3	35.2	30.8
Apparent Density	g/cc	0.72	0.75	0.69	0.78	0.83	0.72
Grid, Type		8x8 LA			8x8 LA		
Weight	g	15.6	16.0	15.2	15.7	16.7	15.2
Form		3.60x3.00x0.25"					
<u>Experimental Conditions</u>							
Sinter Temp., Initial	°F	1700	-	-	-	-	-
" " Average	°F	1659	1685	1639	1655	1667	1628
Sintering Time	min.	10	-	-	-	-	-
<u>Results</u>							
Thickness	mm.	4.28	4.42	4.16	4.47	4.72	4.27
Shrinkage - % of Compact		32.7	34.5	30.4	29.6	32.1	25.7
Plate Area	sq.in	10.51			10.51		
<u>Calculated Results</u>							
Plate Volume	sq.in.-mm	44.98			46.98		
Pore "	cc	23.73	24.65	22.92	24.71	26.39	23.69
% of Apparent Sinter Vol.		86.9	87.6	86.3	86.5	87.1	85.8
% of Final Plate Volume		81.8	82.6	81.0	81.6	82.4	80.9
Unit Pore Vol, cc/sq in-mm		0.53	-	-	0.53	-	-
" Plate Wt, g/sq in-mm		1.030			1.043		
" Powder "		0.683			0.710		
" Grid "		0.347			0.333		



8. Appendix (Contd.)

Table No. III

**Sintering; Perforated Rolled and Electroformed Nickel  
Sheet as Grids**

Experiment No.	S	449-50	451-2	453-4	455-6	457-8	463	464
Date	1947		11/26-	- - -	-11/28-	- - -	-12/3-	- - -
Record	Vol	IV, 173-7	- - -	- - -	- - -	- - -	VIII,	110- - -
Operator		L. Fay & W. W. Oppenheim - - - - -						
<u>Compact Preparation</u>								
Powder, Type		P23-2	- - -	- - -	- - -	- - -	- - -	- - -
Weight Sintered	g	45.50	47.40	45.75	45.20	44.54	45.77	23.42
Apparent Density	g/cc	1.03	1.08	1.04	1.04	1.05	nd	nd
Grid, Type		8x8L Perforated sheet, See Notes						
Weight	g	14.52	5.18	4.46	8.89	12.78	4.13	4.09
Form, 3.60x3.00", Depth		0.25 inch	- - -	- - -	- - -	- - -	- - -	0.125
<u>Experimental Conditions</u>								
Temp, Initial	°F	1715-	- - -	- - -	- - -	- - -	1720	1715
" Final	°F	1670	1677	1662	1665	1665	1675	1655
" Average	°F	1654	1660	1650	1652	1653	1661	1647
Sinter Time	min	10	- - -	- - -	- - -	- - -	- - -	- - -
<u>Results</u>								
<u>Dimensions</u>								
Shrinkage in Width	%	1.58	1.69	1.53	1.53	1.53	0.99	0.99
" " Height	%	2.04	1.91	2.04	2.04	1.91	1.31	1.31
Thickness	mm	5.28	5.32	5.27	5.27	5.28	5.21	2.64
Shrinkage - % of Compact		16.8	16.2	17.0	17.0	16.8	18.0	16.8
Ratio, Form/Plate Thick.		1.20	1.20	1.20	1.20	1.20	1.22	1.20
Plate Area	sq.in	10.41	- - -	- - -	- - -	10.43	10.51-	- -

Notes (1) Perforated grids used throughout. For plates 451-458 holes were 3/8 inch in diameter with the centers of alternate rows staggered. Lugs were cut out of the solid sheet, integral with the grid. For plates 463 and 464, the holes were one-half inch in diameter. The grid size was made to allow 1/8 inch clearance on all sides.

For 449 & 450, 8x8 wire cloth, for comparison

" 451 & 452, International Nickel Co., 0.005 inch electroformed Ni sheet

" 453 & 454 " " " 0.005 " rolled sheet, A nickel

" 455 & 456 " " " 0.010 " " " " "

" 457 & 458 " " " 0.015 " " " " "

" 463 & 464 Chromium Corp. of Am. 0.009 " electroformed Ni sheet

## 8. Appendix (Contd.)

Table No. IV

## Sintering; Effect of Thin Sintered Sheet as Grid

Experiment No.	S	371	372	447	448	462
Date	1947	8/27	- - - -	11/10	- - - -	12/1
Record	Vol	V, 82	- - - -	VIII 75, 76	- - - -	IV, 180
Operator		P.S.	- - - -	L.F.	- - - -	- - - -
<u>Compact Preparation</u>						
Powder, Type		P21	- - - -	P21-3	- - - -	P23-2
Weight Sintered	g	n.d.	n.d.	50.36	51.26	49.02
Apparent Density	g/cc	"	"			
Grid, Type		See Notes below - - - - -				
Weight	g	7.3	5.6	nd	14.18	8.07
Form		3.6x3.0x0.125	3.60x3.0x0.25	- - - -	- - - -	- - - -
<u>Experimental Conditions</u>						
Temp., Initial	°F	1700	- - - -	1760	- - - -	1720
" Final	°F	- - -	- - -	1750	1755	1645
" Average	°F	1662	1689	- - -	- - -	1645
Sinter Time	min	10	- - - -	15	- - - -	10
<u>Results</u>						
Dimensions						
Shrinkage in Width	%	nd	nd	4.27	1.09	1.97
" " Height	%	"	"	5.12	1.18	1.97
Thickness	mm	"	"	4.55	4.88	5.18
Shrinkage - % of Compact		"	"	28.8	23.2	18.4

Notes (1) Description of Grids Used. All were rolled sintered sheets of thickness and calculated porosity indicated.

S-371 3.6x3.0", 0.007" thick rectangular cut

S-372 3.6x3.0", 0.012" " " "

S-447 3.35x2.75", 0.020" " , 65% porosity, rectangular cut

S-448 " , 0.020" " 43% " " "

S-462 " , 0.020" " 43% " " "

perforated with 3/8" holes, staggering the centers of alternate rows.

(2) Bend test applied to all samples. Broke squarely and no cohesion between powder and presintered thin sheet shown in any case.

8. Appendix (Contd.)

Table No. V Part 1

Sintering of Light Carbonyl Powder  
Effect of Sintering Time from 3 to 15 minutes

Experiment No.	S	465	466	467	468	469	470
Date	1947	12/1-12/2	-	-	-	-	-
Record	Vol.VIII	97-99	-	-	-	-	-
Operator		L.F. & W.W.O.					
<u>Compact Preparation</u>							
Powder, Type		-	-	-P23-2	-	-	-
Weight Sintered	g	45.19	45.48	n.d.	44.85	43.84	45.30
Apparent Density	g/cc	1.06	1.06	"	1.05	1.03	1.06
Grid, Type		8x8 LA	-	-	-	-	-
Weight	g	14.56	14.52	14.40	14.45	13.83	14.50
Form		3.60"x3.00"x0.250"	-	-	-	-	-
<u>Experimental Conditions</u>							
Temp., Initial		1720	-	-	-	-	-
" Final		1635	1625	1620	1620	1630	1635
" Average		1668	1665	1655	-----	1656	-----
Sinter Time	min.	3-	-	4	-	5	-
<u>Results</u>							
<u>Dimensions</u>							
Shrinkage in Width	%	1.0	1.31	nd	1.0	1.42	1.42
" " Height	%	0.92	1.18	-	0.92	1.57	1.44
Thickness	mm	5.97	5.92	5.79	5.79	5.69	5.69
Shrinkage - % of Compact		6.0	6.8	8.8	8.8	10.4	10.4
Ratio, Form: Plate Thickness		1.063	1.072	1.098	-----	1.117	-----
Plate Area	sq.in.	10.59	10.53	nd	10.59	10.47	10.49
<u>Calculated Results</u>							
Plate Volume	sq.in.-mm.	63.3	62.4	nd	61.31	59.6	59.6
Pore Volume	cc	33.95	33.35	nd	32.78	31.86	31.66
% of Apparent Sinter Volume		86.5	86.4	nd	86.3	86.3	85.8
% of Final Plate Volume		83.1	82.9	nd	82.8	82.9	82.3
Unit Pore Volume, cc/sq.in.-mm.		0.536	0.534	nd	0.534	0.534	0.534
" Plate Weight g/ "		0.944	0.962	nd	0.967	0.968	1.129
" Grid " g/ "		0.230	0.232	nd	0.236	0.232	0.273
" Powder " g/ "		0.714	0.730	nd	0.732	0.736	0.856

Notes (1) Up to plate No. 474, plate lugs are 3/8 inch wide;  
From 474 to 490, " " " 0.23 " "

8. Appendix (Contd.)

Table No. V Part 2

Sintering of Light Carbonyl Powder  
Effect of Sintering Time from 3 to 15 minutes

Experiment No.	S	471	472	473	474	475	476	477
Date	1947	12/2-12/3	-	-	-	-	-	-
Record	Vol.VIII	pp. 100-103	-	-	-	-	-	-
Operator		L.F. & W.W.O.	-	-	-	-	-	-
<u>Compact Preparation</u>								
Powder, Type		P23-2	-	-	-	-	-	-
Weight Sintered	g	45.65	45.08	44.98	44.92	44.79	44.26	45.67
Apparent Density	g/cc	1.07	1.05	1.05	1.05	1.05	1.04	1.07
Grid, Type		8x8 LA	-	-	-	-	-	-
Weight	g	15.0	14.43	14.38	14.86	14.87	14.87	14.92
Form		3.60"x3.00"x0.25"	-	-	-	-	-	-
<u>Experimental Conditions</u>								
Temp, Initial	°F	1720-	-	-	-	-	1715	-
" Final	°F	1635-	-	-	1655	1645	1645	-1650
" Average	°F	1647	-	-	1656	1647	1645	1644
Sinter Time	min.	6	-	-	7	-	8	-
<u>Results</u>								
<u>Dimensions</u>								
Shrinkage in Width	%	1.10	1.42	1.42	1.10	1.53	1.53	1.53
" " Height	%	1.05	1.57	1.57	1.31	1.97	1.97	1.97
Thickness	mm	5.64	5.56	5.46	5.46	5.33	5.33	5.26
Shrinkage - % of Compact		11.2	12.4	14.0	14.0	16.0	16.0	17.2
Ratic, Form: Plate Thickness		1.123	1.141	1.162	1.162	1.191	1.191	1.209
Plate Area	sq.in.	10.56	10.47	10.47	10.54	10.42	-	-
<u>Calculated Results</u>								
Plate Volume	sq.in.-mm.	59.60	58.30	57.20	57.20	55.6	55.5	54.8
Pore Volume	cc	31.52	30.77	30.11	30.33	29.05	29.11	28.47
% of Apparent Sinter Vol.		85.6	85.4	85.2	85.4	84.7	84.9	84.4
% of Final Plate Vol.		81.9	81.9	81.6	81.8	81.0	81.2	80.6
Unit Pore Volume, cc/sq.in.-mm.		0.529	0.528	0.526	0.527	0.523	0.524	0.520
" Plate Weight	g/ "	1.019	1.021	1.037	1.038	1.074	1.068	1.105
" Powder	g/ "	0.252	0.248	0.251	0.258	0.267	0.267	0.272
" Grid	g/ "	0.767	0.773	0.786	0.780	0.807	0.801	0.833

8. Appendix (Contd.)

Table No. V Part 3

## Sintering of Light Carbonyl Powder

Effect of Sintering Time from 3 to 15 minutes

Experiment No.	S	478	479	480	481	482	483	484
Date	1947	12/3	-	-	-	-	-	-
Record	Vol.	VIII	103-106	-	-	-	-	-
Operator		L. F. & W.W.O.	-	-	-	-	-	-
<u>Compact Preparation</u>								
Powder, Type		P23-2	-	-	-	-	-	-
Weight Sintered	g	43.39	45.31	45.11	43.76	45.37	45.26	45.18
Apparent Density	g/cc	1.02	1.06	1.06	1.02	1.06	1.06	1.06
Grid, Type		8x8 LA	-	-	-	-	-	-
Weight	g	14.87	15.04	14.78	14.76	14.76	14.72	14.82
Form		3.60"x3.00"x0.25"	-	-	-	-	-	-
<u>Experimental Conditions</u>								
Temp, Initial	°F	1715	-	-	-	-	-	-
" Final	°F	1660	1670	-	1680	-	1685	1695
" Average	°F	1649	1650	1652	1654	1654	1655	1661
Sinter Time	min.	9	10	-	11	-	12	-
<u>Results</u>								
<u>Dimensions</u>								
Shrinkage in Width	%	1.64	-	-	-	-	1.53	1.64
" " Height	%	2.10	-	-	-	-	1.97	-
Thickness	mm.	5.21	5.15	-	5.05	5.08	5.05	4.95
Shrinkage-% of Compact		18.0	18.8	18.8	20.4	20.0	20.4	22.0
Ratio, Form: Plate Thickness		1.219	1.230	1.230	1.257	1.250	1.257	1.281
Plate Area	sq.in	10.39	-	-	-	-	10.42	10.41
<u>Calculated Results</u>								
Plate Volume	sq.in-mm	54.1	53.5	53.5	52.5	52.8	52.6	51.5
Pore Volume	cc	28.32	27.68	27.73	27.22	27.23	27.14	26.42
% of Apparent Sinter Volume		84.9	84.1	84.2	84.4	83.8	83.7	83.4
% of Final Plate Volume		81.1	80.2	80.3	80.4	79.9	79.9	79.4
Unit Pore Volume, cc/sq.in-mm		0.524	0.518	0.518	0.519	0.516	0.516	0.514
" Plate Weight	g/ "	1.078	1.127	1.119	1.115	1.138	1.140	1.166
" Powder "	g/ "	0.275	0.281	0.276	0.281	0.280	0.280	0.288
" Grid "	g/ "	0.803	0.846	0.843	0.834	0.858	0.860	0.877

8. Appendix (Contd.)

Table No. V Part 4

## Sintering of Light Carbonyl Powder

## Effect of Sintering Time from 3 to 15 minutes

Experiment No.	S	485	486	487	488	489	490
Date	1947	12/3-12/4	-	-	-	-	-
Record	Vol.VIII	pp. 107-9	-	-	-	-	-
Operator		L. F. & W.W.O.	-	-	-	-	-
<u>Compact Preparation</u>							
Powder, Type		P23-2	-	-	-	-	-
Weight Sintered	g	44.10	45.87	45.44	44.95	45.41	45.70
Apparent Density	g/cc	1.06	1.07	1.06	1.05	1.06	1.07
Grid, Type		8x8 1A	-	-	-	-	-
Weight	g	14.90	14.69	14.81	14.75	14.79	14.81
Form		3.60"x3.00"x0.25"	-	-	-	-	-
<u>Experimental Conditions</u>							
Temp, Initial	°F	1715	-	-	-	-	-
" Final	°F	1695	1705	1700	1700	1710	1705
Average	°F	1660	1670	1660	1662	1665	1660
Sinter Time	min.	13	-	14	-	15	-
<u>Results</u>							
<u>Dimensions</u>							
Shrinkage in Width	%	1.75	1.53	1.97	2.08	1.75	1.86
" " Height	%	2.34	2.10	2.49	2.62	2.49	2.49
Thickness	mm	4.93	5.00	4.98	4.83	4.83	4.88
Shrinkage-% of Compact		22.4	21.2	21.6	24.0	24.0	23.2
Ratio, Form: Plate Thickness		1.289	1.270	1.274	1.316	1.316	1.302
Plate Area	sq.in.	10.36	10.41	10.32	10.29	10.34	10.33
<u>Calculated Results</u>							
Plate Volume	sq.in.-mm.	51.10	52.10	51.40	49.70	50.00	50.50
Pore Volume	cc	26.24	26.69	26.31	25.29	25.39	25.66
% of Apparent Sinter Vol.		83.8	83.4	83.4	82.9	82.8	82.9
% of Final Plate Volume		79.7	79.6	79.4	78.8	78.7	78.9
Unit Pore Volume, cc/sq.in.-mm.		0.514	0.513	0.512	0.509	0.508	0.509
" Plate Weight g/ "		1.154	1.163	1.171	1.200	1.204	1.199
" Powder " g/ "		0.292	0.282	0.288	0.296	0.296	0.293
" Grid " g/ "		0.862	0.881	0.883	0.904	0.908	0.906

8. Appendix (Contd.)

Table No. VI

Sintering; Routine Production of 10.5 sq. in. Plates

from Light Carbonyl Powder, P23-2

Experiment No.	S	498-505	506-512
Date	1947	12/10	12/11-12/12
Record	Vol.	VIII 112-4	VIII 116, VII18-9
Operator		L.F. & W.W.O.	-----
<u>Compact Preparation</u>			
Powder, Type		P-23-2	-----
Weight Sintered	g	15.74	48.15
Apparent Density	g/cc	1.02-1.14	1.13
Grid, Type		8x8 Wide Lug	8x8 1A
Weight	g	22.05	15.57
Form, 3.6x3.0, Depth	inch	0.094	0.250
<u>Experimental Conditions</u>			
Temp, Initial	°F	1715	1715
" Final	°F	1635-1675	1640-1670
Time of Sinter	min	10	10
<u>Results</u>			
<u>Dimensions</u>			
Shrinkage in Width	%	1.10	1.44
" " Height	%	1.31	1.97
Thickness	mm	2.08-2.16	5.05-5.26
Shrinkage-% of Compact		9.6-12.8	31.8-33.1
Ratio, Form: Plate Thickness		1.103-1.145	1.208-1.257
Plate Area	sq.in.	10.55	10.43
<u>Calculated Results</u>			
Plate Volume	sq.in.-mm	22.35	53.71
Pore "	cc	10.16-10.79	27.41
% of Apparent Sinter	Vol.	84.4-85.9	83.0
% of Final Plate	"	71.8-73.4	79.0
Unit Pore Volume, cc/sq.in.-mm		0.47	0.51
" Plate Weight	g/ "	1.693	1.183
" Powder "	g/ "	0.705	0.894
" Grid "	g/ "	0.988	0.289

Notes (1) Plates 498-505 were provided with perforated nickel sheet grids of the same stock as used in the early experiments. The rectangular portion was 3-23/64 x 2 3/4" with a lug in one corner 1-1/2" x 1"

(2) Lugs for plates 506-512, 0.23 inch wide

8. Appendix (Contd.)

Table No. VII

Sintering of Light Carbonyl Nickel Powder  
10.7 SQ. IN. PLATES from 3 piece Form

Experiment No.	1	2	3	4-5	6-19	20	21	22
Date	9/25	9/26	9/29	10/2	10/13-10/24	10/13-10/24	11/20	11/20
Record	2	3	4	10-11	19-42	19-42	81	81
Operator	P.S.			L.F.				
Compact Preparation								
Powder, Type	P21-1				P21-2			P22
Weight Sintered	37.62	37.03	34.48	36.74	37.40	41.41	42.00	55.53
Grid, Type	8x8 LA							
Weight	17.5	17.45	17.0	16.9	16.2	15.9	16.0	16.0
Form	3 piece, FM-1, 3x3-52/64x0.25							
Experimental Conditions								
Temp, Initial	1810	1760	1810	1765	1810	1805	1800	
" Final						1710	1770	
" Average	1717	1672	1722	1678	1726			
Sinter Time	10	15	15	15	14	15	16	14
Results								
Thickness	5.13	4.70	4.37	4.60	4.43	4.67	4.52	6.04
Shrinkage-% of Compact	19.2	26.0	31.2	27.6	30.4	26.4	28.8	4.8
Shrinkage in Width	1.24	1.34	1.75	1.44	1.75	nd	nd	0.83
" " Height	1.97	2.23	2.36	2.49	2.23	nd	nd	0.79
Ratio, Form: Plate Thickness	1.238	1.352	1.454	1.383	1.438	1.360	1.406	1.051

See Table XIII, 15 for Notes



8. Appendix (Contd.)

Table No. VIII

## Sintering of Light Carbonyl Nickel Powder

12.5 sq. in Plates from 3 piece Form

Experiment No.	FM-2	1	3	6	2,4,5,7	8-9	10-19	20
Date	1947	9/25	9/29	10/1	- - - -	-10/2	10/9-22	10/23
Record	Vol.VIII	2/10	- - - -	- - - -	- - - -	- - - -	14-39	40
Operator		P.S	- - - -	- - - -	- - - -	L.F.	- - - -	- - - -
<u>Compact Preparation</u>								
Powder, Type		P21-1	- - - -	- - - -	- - - -	- - - -	P21-2	- - - -
Weight Sintered	g	42.55	42.09	41.42	42.85	41.59	43.70	44.69
Grid, Type		8x8	LA	- - - -	- - - -	- - - -	- - - -	- - - -
Weight	g	18.5	18.5	18.5	18.5	18.3	18.4	18.4
Form		3 piece, FM-2, Brx 3-27/64x3-53/64x0.25						
Scraper, Depth	inch	0.190	0.205	0.195	0.195	0.200	- - - -	- - - -
<u>Experimental Conditions</u>								
Temp, Initial	oF	1710	1800	1765	- - - -	- - - -	1810	1810
" Final	oF	- - -	- - -	- - -	- - -	- - -	1745	1760
" Average	oF	1637	1712	1667	1679	1680	1723	- - -
Sinter Time	min	10	15	12	15	13	14	18
<u>Results</u>								
Thickness	mm	5.64	4.37	4.72	4.63	4.52	4.46	4.14
Shrinkage-% of Compact		11.2	31.2	25.6	26.8	28.8	30.8	34.8
Shrinkage in Width %		1.34	2.17	1.65	1.91	1.76	1.86	nd
" " Height %		1.50	2.19	1.96	2.16	2.07	2.24	nd
Ratio, Form: Plate Thickness		1.126	1.454	1.344	1.366	1.404	1.419	1.533

See Table XIII, 15 for Notes

## 8. Appendix (Contd.)

Table No. IX

## Sintering of Light Carbonyl Nickel Powder

14 sq. in. Plates from 3 piece form

Experiment No.	FM-3	1	2	3	4-9	10	11-116	117-123	124-143	128-134
Date	1947	9/25	9/26	9/29	9/29-10/1	10/9	10/9-11/5	11/5-11/6	11/6-11/7	
Record	Vol. VIII	2	3	4	5-8	14	14-66	66-67	69-74	
Operator	P.S.				L.F.					
Compact Preparation - No Averaged	1	1	1	6		1	106	7	14	6
Powder, Type	P21-1					P21-2		P21-3		
Weight Sintered	47.45	48.44	43.94	46.52		49.64	48.16	49.92	51.12	52.00
Grid, Type	8x8 LA									
Weight	20.8	20.8	21.2	20.8		20.5	19.8	18.8	18.8	10.15
Form	3 piece, FM-3	3-27/32x3-53/64x0.25"								
Experimental Conditions										
Temp, Initial	1715	1760	1810	1760		1760	1810			
" Final							1740		1772	1758
" Average	1626	1674	1701	1678		1684	1719			
Time of Sinter	10	15	10	15		14	14-15	15	14	14
Results										
Thickness	5.67	5.21	4.65	4.54		4.67	4.39	4.45	4.54	4.43
Shrinkage-% of Compact	10.8	18.0	26.8	28.5		26.4	30.8	30.0	28.5	30.4
Shrinkage in Width	0.72	0.93	0.83	1.16		0.93	1.11	nd	nd	nd
" Height	2.35	2.56	2.25	2.69		2.68	2.68	nd	nd	nd
Ratio, Form: Plate Thickness	1.121	1.220	1.368	1.400		1.359	1.449	1.429	1.400	1.431
Calculated Results										
Pore Volume	43.48	38.95	35.50	33.21		33.87	32.18	nd	nd	nd
% of App. Powder Volume	88.8	87.3	87.3	86.0		85.5	84.9			
% of Final Plate Volume	84.5	83.2	82.9	81.4		81.0	80.1			

See Table XIII, 15 for notes

### 8. Appendix (Contd.)

Table No. X

#### Sintering on Light Carbonyl Nickel Powder

#### Tests on Grab Samples from 1000 lb. lot

Experiment No.	FM-3	144	147	148	149	150	151
Date	1947	11/10	11/24	-	-	-	-
Record	Vol.VIII	78	83	-	-	-	-
Operator		L. F. & W.W.O.					
<u>Compact Preparation</u>							
Powder Type		P23-1	P23-2	P23-5	P23-6	P23-7	P21-3
Treatment		a.r.	Grab samples	-	-	-	-200
Weight Sintered	g	66.51	64.71	64.74	57.44	54.28	51.51
Apparent Density-Scott	g/cc	0.79	0.88	0.88	0.73	0.70	0.53
App. Density in Form	"	1.14	1.12	1.12	0.99	0.94	0.89
Grid, Type		8x8 LA	-	-	-	-	-
Weight	g	10.3	17.14	17.50	17.13	17.53	17.61
Form		3 piece	FM-3, 0.25 inch cavity				
<u>Experimental Conditions</u>							
Temp, Initial	OF	1815	1800	-	-	-	-
" Final	OF	1790	1750	1770	1760	1755	1765
Sinter Time	min	15	14	-	-	-	-
<u>Results</u>							
Shrinkage in Width	%	2.48	3.20	3.52	3.10	2.90	4.04
" " Height	%	1.84	3.07	3.47	2.97	2.97	4.09
Thickness	mm	4.72	5.15	4.92	4.92	4.90	4.66
Shrinkage-% of Compact		25.6	18.8	22.4	22.4	22.8	26.4
Ratio, Form: Plate Thickness		1.343	1.231	1.288	1.288	1.296	1.359
<u>Calculated Results</u>							
Plate Volume	sq.in.-mm	63.36	70.76	67.06	67.09	67.15	62.30
Pore Volume	cc	33.36	35.76	33.37	34.40	34.90	32.12
% of Apparent Sinter	Vol.	81.0	82.6	81.6	83.6	84.7	84.4
% of Final Plate Volume		78.9	79.1	78.0	80.5	81.0	80.3
Unit Pore Volume, cc/sq.in.-mm		0.526	0.505	0.497	0.512	0.519	0.515
" Plate Weight	g/sq.in.-mm	1.211	1.153	1.227	1.111	1.069	1.110
" Grid	g/ "	0.163	0.242	0.261	0.255	0.261	0.283
" Powder	g/ "	1.048	0.911	0.966	0.856	0.808	0.827

Table No. XI

Effect of Sintering Time from 5 to 15 minutes

3 piece FM-3 Form; P23-2 Light Carbonyl Ni Powder, Unscreened  
8x8 wire cloth grid, 0.028" wire, lug attached, annealed

Experiment No.	152	153	154	155	156	157	158	159	160	161	162
Date	1947	11/25-11/26									
Record	Vol.	VIII 85-95									
Operator	L. F. & W.W.O.										
Compact Preparation											
Powder Sintered											
Grid Wt	62.85	65.98	64.79	65.27	63.11	62.67	61.59	64.13	63.91	62.16	62.87
Apparent Density	17.15	17.49	17.13	16.99	17.01	17.39	17.38	17.15	16.68	16.63	16.78
Experimental Conditions	g/cc	1.09	1.14	1.12	1.13	1.10	1.09	1.07	1.11	1.08	1.09
Temp, Initial	1805										
" Final	1700	1720	1700	1715	1720	1700	1710	1740	1750	1760	1765
" Average	1730	1737	1708	1717	1711	1689	1694	1710	1714	1718	1719
Sinter Time	5	6	7	8	9	10	11	12	13	14	15
Results											
Shrinkage in Width	1.45	1.76	1.76	1.55	1.86	2.38	2.27	2.68	2.38	2.38	2.79
" " Height	1.67	1.67	1.88	1.56	1.98	2.60	3.12	2.71	2.50	2.50	3.02
Thickness	5.94	5.79	5.66	5.56	5.41	5.35	5.23	5.28	5.05	4.86	4.90
Shrinkage	6.4	8.8	10.8	12.4	14.8	15.6	17.6	16.8	20.4	23.2	22.8
Ratio, Form; Plate Thick.	1.069	1.098	1.121	1.141	1.173	1.184	1.212	1.201	1.257	1.303	1.295
Calculated Results											
Pore Volume	43.95	42.61	41.49	40.81	39.31	38.22	37.06	37.24	35.48	34.00	33.86
% of Apparent Vol.	85.6	85.0	84.4	84.4	84.1	84.0	83.7	83.3	82.9	82.4	82.3
% of Plate Volume	82.5	81.8	81.5	81.3	81.2	80.9	80.3	80.0	79.4	79.0	79.0
Plate Vol.	83.3	82.0	80.1	79.0	76.4	74.6	72.5	73.3	70.5	68.0	67.7
sq.in.-mm											
cc/ " Unit Pore Vol.	0.527	0.521	0.517	0.517	0.515	0.513	0.511	0.509	0.504	0.500	0.500

See Table XIII, 17 for Note

8. Appendix (Contd.)

Table No. XII

## Sintering of Light Carbonyl Nickel Powder

## Routine Production of 14 sq. in. Plates in 3 piece Form

Experiment No.	FM-3	163-182		183-202	203-20
Date	1947	12/11-12/12		12/12-12/18	12/18
Record	Vol. VIII	115-123		123-129	129-13
Operator		L. F. & W.W.O.			
Compact Preparation		Average High Low		Average	Average
Powder, Type		P23-2 unscreened			
Weight Sintered	g	54.84	59.81	51.02	55.54
Apparent Density	g/cc	1.42			52.27
Grid, Type		8x8 LA			
Weight	g	16.79	18.86	16.22	16.66
Form		3 piece FM-3, 0.203 inch cavity			19.97
Experiment Conditions					
Temp., Initial	°F	1717	1720	1715	1732
" Final	°F	1629	1640	1615	1645
" Average	°F	1620	1625	1609	1636
Sinter Time	min.	10			
Results					
Shrinkage in Width	%	2.42	3.10	1.65	2.42
" " Height	%	2.48	3.18	1.85	2.24
Thickness	mm.	4.57	4.80	4.34	4.59
Shrinkage, % of Compact		11.37	15.74	6.89	10.88
Ratio, Form; Plate Thickness		1.128	1.072	1.190	1.125
Calculated Results					
Pore Volume	cc	32.19	34.00	30.56	
% of Apparent Sinter Vol.		83.5			
% of Final Plate Volume		79.8			
Plate Volume	sq. in.-mm	63.75			
Unit Pore Volume, cc/sq. in.-mm		0.504			
Unit Plate Weight, g/sq. in.-mm		1.122			
" Powder	"	0.858			
" Grid	"	0.264			

See Table XIII, 18 for Notes

8. Appendix (Contd.)

Table No. XIII

Notes on Tables I-XII

General

1. Compact Preparation. All S experiment plates were made in the two piece form, previously described, with the bottom piece having a cavity 3.60x3.00 inches and provided with a lug cavity at one corner. The depth of the cavity was either 1/8 or 1/4 inch. FM plates were made in a three piece mold, for which a complete description will be given in the annual report, with height varying as indicated in the table. The center piece is a spacer whose thickness can be varied, thus allowing for desired changes in plate thickness.

2. Powder Type. All experiments described in this report were made on light carbonyl powder. The powder numbers and descriptions will be found in Table No. 1.

3. Weight Sintered. The weight of powder sintered in any plate is found by subtracting the grid weight from the final plate weight. The latter was determined after suitable trimming, especially to remove the powder sintered in the lug cavity, thereby permitting calculations on the basis of the rectangular plate with rounded corners.

4. Apparent Density under the heading of Compact Preparation applies to the apparent density of filling the form cavity with powder, correcting for the volume occupied by the grid. The true volume of nickel powder is based on a density of 8.6 g per cc.

5. Grid, Type. In general, the use of the 8x8 mesh nickel wire cloth grid with spot-welded lug has been adopted. The grids are annealed at 1700°F for 10 minutes in the sintering furnace in a protective gas atmosphere. Except, where noted, the wire cloth is made from 0.028 inch (diameter) wire. For S plates the lug width has been increased from 1/4 to 3/8 inch width while for the FM plates the width was standardized at 3/8 inch, and 0.031 inch thickness. The standard grids are designated as 8x8 IA.

8. Appendix (Contd.)

Table No. XIII, contd.

6. Experimental Conditions All plates were made in the Rockwell furnace as described, using a protective gas atmosphere of dissociated ammonia at a flow of 1.5 CFM, as indicated by a Rotameter flowmeter calibrated for air flow. The dissociator is operated at 1700°F with no provisions for testing gas for residual oxygen, moisture, etc.

7. Temperature The initial temperature (same as nominal, previously reported) is the furnace temperature prior to opening the front door and inserting the sample. To obtain a more uniform average temperature, the procedure was standardised by placing the sample into the furnace one-half minute after the circuit breaker (for the heating coils) opens. Readings of the thermocouple in the gas space were made at two minute intervals and averaged. It was noted that the average temperature for a given series increased during the day; the furnace temperature was lowered overnight and over weekends.

8. Sintering Time refers to the time after the inserting the form into the furnace to the time it is pushed from the high temperature zone into the cooling chamber, the latter generally requiring about 20 minutes.

9. Results. The plates are examined and measured. Shrinkages are calculated from the difference between the measured dimensions and the specified form dimensions, and expressed in terms of percentages of the form dimensions.

10. Calculated Results. These values are specifically indicated so that it will be clear at a later date when it is proposed to measure these values quantitatively by suitable experimental procedures.

11. Apparent Sinter Volume represents the volume found by subtracting the grid volume from the final plate volume. This figure is important in expressing the porosity for comparative purposes since it represents the actual porosity of the sintered powder. It is obvious that the porosity of any plate can be varied by changing the grid weight and volume.

8. Appendix (Contd.)

Table No: XIII, contd.

12. Pore Volume. Subtracting the true volume of the nickel powder, taken at a density of 8.6 g. per cc., from the apparent sinter volume, see note 11 above, gives the pore volume directly. The use of the density 8.6 instead of the reported 8.9 g. per cc. gives a slightly lower pore volume and percentage porosity. These two values represent the range of values reported for nickel metal. The use of the low value is justified on the basis of the reported variations of density with various liquids related to the wetting characteristics of the liquid used for the determination. On the other hand it is conceivable that a small proportion of closed pores, useless for impregnation may be formed in the operation.

13. Plate Volume has been expressed in terms of a mixed unit, sq. in.-mm, as a result of expressing area in sq. in. and plate thickness in mm., both convenient, inasmuch as in terms of this unit, the plate weights have been running about 1 gram, plus or minus, and pore volume about 0.5 cc, plus or minus. It provides a ready comparison for plates of various size and an easy figure for use in design.

14. Plate Storage. Aside from keeping the plates clean and uncontaminated, and cataloging properly, no special precautions have been taken to keep in a dry or other special atmosphere up to the time that the plates are impregnated with active mass.

15. Special Notes. Tables No. VII-IX

(1) Beginning with Expt. No. 4 in each of the series FM1, FM2, and FM3, the lug cavity was filled with powder to prevent movement of the grid during handling of the form.

(2) For the three piece form used, the cavity was filled to 0.0025 inch above the level to which the top or cover plate descended on proper fitting of the cover plate. Beginning with Expt. 2 in each series, the form with the green compact was inverted after assembly and before sintering.



8. Appendix (Contd.) Table No. XIII, contd

15. Cont'd (3) Plate FM3-38 was made up with powder which had been dried for 12 hours at 110°C in an Isotemp oven, cooled and kept in a desiccator till used.

(4) Prior to sintering plate FM3-53 the protective gas atmosphere was shut off and was inadvertently left off. Apparently the residual hydrogen atmosphere was sufficient to prevent oxidation of the nickel.

(5) Table No. IX, plates 128, 129, 130, 131, 132 and 134 were made up with 8x8 mesh grids, the wire cloth being woven from 0.020 inch dia. wire. The pattern of spot-welding the wire cloth was selected to give a rigid framework. This use of lighter wire results in a saving of 37% of the weight of nickel.

16. Special Notes, Table X

(1) Sample P23 represents a 1000 lb. lot shipment of carbonyl nickel powder, received in four steel drums. P23-1 was the sample shipped by the manufacturer and stated to be representative of the entire shipment. P23-2, P23-5, P23-6, P23-7 represent grab samples taken from the drums for comparative purposes. The differences in apparent density and shrinkage indicated the need for careful sampling. P23-2 drum (Figure 2 indicates manufacturers' drum number) was quartered by means of a Jones riffle to obtain a representative sample.

17. Special Notes, Table XI

(1) New spacers were provided at the start of FM3-152 due to wear of spacers by brass powder leveling tool. A steel cover plate was also provided at this point to reduce or eliminate this wear.

18. Special Notes, Table XII

(1) Beginning with FM3-203, the wire cloth grid was cut somewhat larger, that is 3.5 x 3.5 inches, than used in the previous runs as can be judged by the grid weights.

(2) Note decrease in time factor to 10 minutes

Table No. XIV Part 1

Atmospheric Impregnation of Negative Plates  
Effect of Time at Room Temperature, 2 mm. Plates

Experiment No.	Im	127	129	131	133	141	145	143	135	137	139
Date	1947	9/2	9/2	9/2	9/2	9/5	9/8	9/8	9/3	9/3	9/5
Record	IV	100	101	102	103	120	122	121	104	107	108
Plate Description	No-X	276	292	293	295	305	316	306	302	303	304
Powder	Type	P-21									
Grid	"	8x8L									
Sinter Temp.	of	1700									
Thickness	mm	2.2	2.0	2.2	2.1	2.0	2.3	2.2	2.2	2.2	2.1
Pore Volume	cc	11.58	10.25	11.57	10.91	10.26	12.18	11.55	11.56	11.52	10.95
Volume, sq. in x mm		23.15	21.02	23.15	22.10	21.02	24.20	23.15	23.15	23.15	22.10
Impregnation, Cycles	No.	1	1	1	1	1	1	1	1	1	1
Solution		Ca(NO <sub>3</sub> ) <sub>2</sub>									
Conditions		All at Room Temperature									
Time, Min., Hr <sup>o</sup>		5'	10'	15'	30'	1°	2°	3°	6°	24°	48°
Pressure			Atmospheric Pressure								
Acidity-Initial		3.45	3.34	3.18	3.61	3.44	3.05	3.05	3.41	3.59	n.d.
Cathodic Polarization											
Current	Amps.	20	20	20	20	20	20	20	20	20	20
Time	Min.	30	30	25	29	20	25	20	20	20	20
Volts, Initial		0.62	0.55	0.52	0.51	0.59	0.58	0.69	0.68	0.64	0.62
Final		1.01	1.99	0.90	0.91	0.88	0.90	0.91	0.89	0.91	0.93
Temp - Initial	°C	89	90	96	96	95	91	90	90	89	89
Washing	Hrs.	3	3	3	3	3	3	3	3	3	3
Drying		Overnight									
Results											
Gain in Wt.-Total	g.	6.17	5.65	5.89	7.01	9.64	9.39	9.78	10.72	10.44	8.44
Equivalent Capacity	A.H.	2.26	2.07	2.16	2.57	3.52	3.44	3.58	3.93	3.83	3.09

8. Appendix (contd.)

Table No. XIV Part 2  
Atmospheric Impregnation of Negative Plates  
Multiple Impregnations

Experiment No. Plate Description	Im	127	129	131	133	141	143	135	137	139
	No-S	276	292	293	295	305	306	302	303	304
<u>Impregnation</u>	No	3	---	---	---	2	---	1	2	---
Cycles										
Conditions										
Temperature										
Pressure										
Time	min.	5	10	15	30	60			-1440	60
Solution										
Acidity										
Cathodic Polarisation										
Current	amps	20								
Time	min.	18 to 30, to final steady voltage								
Volts, Initial		0.62	0.55	0.52	0.51	0.59	0.58	0.69	0.64	0.62
Final		2.14	2.09	2.13	2.29	2.11	2.09	2.17	2.21	2.10
Temp., Initial	°C	91	92	94	92	93	91	91	91	91
Washing	Hrs.	3								
Drying		Overnight								
Results										
Gain in Wt.-Total	g.	17.11	16.59	15.41	17.02	12.58	13.79	12.40	13.65	11.50
Equivalent Capacity	A.H.	6.27	6.07	5.65	6.24	4.61	5.05	4.54	5.00	4.21
Capacity Test										
Test Cell	No.	---	G-1	---	116	94	---	117	---	---

8. Appendix (contd.)

Table No. XV Part 1

## Atmospheric Impregnation of Negative Plates

Effect of Time at Room Temperature, 4 mm. plates

Experiment	Im	128	130	132	134	142	146	144	136	138	140
Date	1947	5/2	9/2	9/2	9/2	9/5	9/8	9/8	9/3	9/3	9/5
Record	IV	100	101	102	103	120	122	121	104	107	108
Plate Description	No-S	288	307	321	322	331	333	332	323	324	325
Powder	Type	P-21									
Grid	"	8x8 L									
Sinter Temp	OF	1700									
Thickness	mm	4.3	4.1	4.2	4.1	4.2	4.0	4.3	4.1	4.2	4.2
Pore Volume	cc	23.90	22.50	23.15	22.49	23.25	21.80	24.00	22.57	23.33	23.26
Volume sq.in. x mm		45.2	43.1	44.2	43.1	44.2	42.1	45.2	43.1	44.2	44.2
Impregnation Cycles	No.	1	1	1	1	1	1	1	1	1	1
Solution		Cd(NO <sub>3</sub> ) <sub>2</sub>									
Conditions		All at Room Temperature									
Time - Min <sup>1</sup> , Hr <sup>o</sup>		5 <sup>1</sup> 10 <sup>1</sup> 15 <sup>1</sup> 30 <sup>1</sup>				1 <sup>o</sup>	2 <sup>o</sup>	3 <sup>o</sup>	6 <sup>o</sup>	24 <sup>o</sup>	48 <sup>o</sup>
Pressure		All at Atmospheric Pressure									
Acidity-Initial		3.45	3.34	3.18	3.61	3.44	3.05	3.05	3.41	3.59	n.d.
Cathodic Polarization											
Current	Amps	20	20	20	20	20	20	20	20	20	20
Time	Min.	30	30	25	29	20	25	20	20	20	20
Volts, Initial		0.61	0.56	0.52	0.54	0.60	0.62	0.72	0.73	0.70	0.66
Final		0.87	0.85	0.84	1.05	0.80	0.91	0.82	0.78	0.83	0.82
Temp - Initial	OC	89	90	96	97	91	91	90	90	89	93
Washing	Hrs.	3	3	3	3	3	3	3	3	3	3
Drying		Overnight									
Results											
Gain in Wt.-Total	g.	14.08	12.13	14.75	15.96	19.81	18.66	18.74	19.25	17.76	18.72
Equivalent Capacity	A.H.	5.16	4.44	5.40	5.84	7.26	6.84	6.87	7.05	6.51	6.86

8. Appendix (contd.)

Table No. XV Part 2

Atmospheric Impregnation of Negative Plates  
Multiple Impregnations

Experiment No. Plate Description	Im No.	128	130	132	134	142	146	144	136	138	140
	No.	283	307	321	322	331	333	332	323	324	325
See also Table XV, Part 1											
<u>Impregnation</u> Cycles	No.	3				2			-1	2	
Conditions		For First Impregnation, see Table XV, Part 1									
Temperature	Room										
Pressure											
Time	Min.	5	10	15	30	60				1440	60
Solution		Atmospheric									
Acidity		Cd(NO <sub>3</sub> ) <sub>2</sub>									
Cathodic Polarization		Initial Acidity adjusted to between 3 to 4 g. HNO <sub>3</sub> per l.									
Current	Amps	20									
Time	Min	18 to 30 to final steady voltage									
Volts, Initial		0.61	0.56	0.52	0.54	0.60	0.62	0.72		0.70	0.66
Final		1.99	2.01	2.17	2.00	2.19	2.03	2.01		2.06	2.00
Temp., Initial	OC	91	92	94	94	91	92	92		92	93
Washing		3 Hours									
Drying		Overnight									
Results											
Gain in Wt.--Total	g.	32.93	31.70	34.26	34.88	28.20	26.57	26.59		27.83	27.22
Equivalent Capacity	A.H.	12.08	11.60	12.54	12.77	10.33	9.73	9.74		10.19	9.98
Capacity Test											
Test Cell	No.			G-2	120		118	119			

# 8. Appendix (cont.)

Table No. XVI

## Atmospheric Impregnation of Negative Plates

Effect of Time from 1 to 6 hours at Room Temperature

Experiment No	Im	147A	147B	147C	147D	147E	147F
Date	1947	11/13	11/13	11/13	11/13	11/13	11/13
Record	Vol. VII	91	92	93	94	95	96
Plate Description	S	440	429	423	407	389	387
Powder	Type	P-21	-	-	-	-	-
Grid	"	8x8L	-	-	-	-	-
Sinter Temp.	°F	1700	-	-	-	-	-
Thickness	mm	4.43	4.43	4.41	4.38	4.38	4.42
Pore Volume	cc	24.49	24.50	24.42	24.54	24.37	24.65
Volume, sq. in x mm		46.60	46.60	46.30	46.10	46.10	46.50
Impregnation, Cycles		1	1	1	1	1	1
Solution		Cd(NO <sub>3</sub> ) <sub>2</sub>	-	-	-	-	-
Conditions		Room Temp	-	-	-	-	-
Time	Hr.	1	2	3	4	5	6
Pressure		Atmospheric	-	-	-	-	-
Acidity-Initial		3.56g. HNO <sub>3</sub> /liter	-	-	-	-	-
Cathodic Polarization							
Current	Amps	21	21	21	21	21	21
Time	Min.	30	33	32	33	34	27
Volts, Initial		1.70	1.74	1.68	1.63	1.66	1.72
Final		2.12	1.96	1.98	2.10	2.16	2.24
Temp, Initial	°C	109	99	103	99	105	106
Washing	Hr.	3	3	3	3	3	3
Drying		Overnight	-	-	-	-	-
Results							
Gain in Wt. Total	g.	19.20	19.67	19.22	17.98	17.31	19.02
Equivalent Capacity	A.H.	7.03	7.20	7.04	6.59	6.34	6.96
Unit Gain		0.784	0.802	0.787	0.734	0.709	0.771
(g per cc Pore Vol)							

8. Appendix (cont.)

Table No. XVII

## Atmospheric Impregnation of Positive Plates

Effect of Time from 1 to 6 Hours at Room Temperature

<u>Experiment No</u>	Im	148A	148B	148C	148D	148E	148F
<u>Date</u>	1947	11/13	11/13	11/13	11/13	11/13	11/13
<u>Record</u>	Vol. VII	91	92	93	94	95	96
<u>Plate Description</u>	S	422	441	438	432	425	424
<u>Powder</u>	Type	P-21	-	-	-	-	-
<u>Grid</u>	"	8 x 8L	-	-	-	-	-
<u>Sinter temp</u>	°F	1700	-	-	-	-	-
<u>Thickness</u>	mm	4.46	4.44	4.45	4.44	4.46	4.44
<u>Pore Volume</u>	c.c.	24.76	24.57	24.57	24.46	24.64	24.60
<u>Volume, sq. in x mm</u>		46.9	46.70	46.80	46.70	46.80	46.70
<u>Impregnation, Cycles</u>		1	1	1	1	1	1
<u>Solution</u>		Ni(NO <sub>3</sub> ) <sub>2</sub>	-	-	-	-	-
<u>Conditions</u>		Room Temp	-	-	-	-	-
<u>Time</u>	Hr.	1	2	3	4	5	6
<u>Pressure</u>		Atmospheric Pressure	-	-	-	-	-
<u>Acidity-Initial</u>		3.51g HNO <sub>3</sub> /Liter	-	-	-	-	-
<u>Cathodic Polarization</u>							
<u>Current</u>	Amps	21	21	21	21	21	21
<u>Time</u>	Min.	30	33	32	33	34	27
<u>Volts, Initial</u>		1.35	1.70	1.39	1.45	1.65	1.40
<u>Final</u>		1.71	1.85	1.76	1.85	2.04	1.89
<u>Temp, Initial</u>	°C	109	99	103	99	105	106
<u>Washing</u>	Hr.	3	3	3	3	3	3
<u>Drying</u>		-	-	-	-	-	-
<u>Results</u>		-	-	-	-	-	-
<u>Gain in Wt.-Total</u>	g.	13.40	13.69	14.68	13.07	14.83	15.38
<u>Equivalent Capacity</u>	A.H.	3.87	3.96	4.24	3.78	4.29	4.44

# 8. Appendix (cont.)

Table No. XVIII

## Atmospheric Impregnation of Positive Plates

Effect of Time from 1 to 5 Days

Experiment No	Im	147G	147H	147I	147J	147K	147L	147M
Date	1947	12/10	12/15					
Record	Vox. IX	39	39	40		41		
Plate Description	S	261	270	287	308	309	349	
Powder	No	P21						
Grid	Type	8 x 8L						
Sinter Temp.	°F	1700						
Thickness	mm	4.25	4.5	4.5	4.3	4.3	2.2	
Pore Volume	cc	23.61	25.19	25.30	23.88	23.85	11.68	
Vol, sq.in x mm		44.7	47.3	47.3	45.2	45.2	23.1	
Impregnation, cycles, No.		1	1	1	1	1	1	
Solution		Ni(NO <sub>3</sub> ) <sub>2</sub>						
Conditions		Atmospheric Pressure - Room Temp.						
Time	Hrs.	24	24	48	48	120	120	
Pressure		Atm.	Atm.	Atm.	Atm.	Atm.	Atm.	
Acidity-Initial		4.03	g HNO <sub>3</sub> per L					
<u>Cathodic Polarization</u>								
Current	Amps.	21	21	21	21	21	21	
Time	Min.	30	30	35	35	30	30	
Voltage-Initial		1.85	1.85	1.6	1.6	1.7	1.7	
Final		2.25	2.25	2.1	2.1	2.1	2.1	
Temp - Initial	°C	107	107	102	102	107	107	
Washing	Hrs.	3	3	3	3	3	3	
Drying		Overnight						
<u>Results</u>								
Gain in Wt. Total	g.	8.72	6.60	13.90	14.52	13.89	8.10	
Equiv. Capacity	A.H.	2.52	1.91	4.02	4.20	4.02	2.34	



# 8. Appendix (contd.)

Table No. XIX  
Atmospheric Impregnation of Positive Plates  
Impregnation at Constant Acidity

Experiment No.	Im	149A	149B	149C	149D	149E	150A	150B	150C	150D	150E
Date	1947	11/19	11/19	11/19	11/19	11/19	11/19	11/19	11/19	11/19	11/19
Record	Vol. VII	107	107	107	107	107	108	108	108	108	108
Plate Type and No.	S	391	383	411	393	403	392	405	406	412	413
Impregnation Cycles	No.	1	1	1	1	1	1	1	1	1	1
Solution		Ni(NO <sub>3</sub> ) <sub>2</sub>					Ni(NO <sub>3</sub> ) <sub>2</sub>				
Conditions		Room Temperature					Room Temperature				
Time	Hr.	1	1	1	1	1	1	1	1	1	1
Pressure		Atmospheric					Atmospheric				
Acidity		Attempted to Hold Constant-Av. 4.14					Attempted to Hold Constant-Av. 3.86				
Cathodic Polarization											
Current	Amps	21	21	21	21	21	21	21	21	21	21
Time	Min	25	25	25	25	25	30	30	30	30	30
Volts, Initial		1.20	1.20	1.20	1.20	1.20	1.30	1.30	1.30	1.30	1.30
Final		2.10	2.10	2.10	2.10	2.10	2.10	2.10	2.10	2.10	2.10
Temp.- Initial	°C	103	103	103	103	103	106	106	106	106	106
Washing	Hr.	3	3	3	3	3	3	3	3	3	3
Drying		Overnight					Overnight				
Results											
Gain in Wt.-Total	g.	4.37	5.27	4.29	4.15	4.96	4.40	5.25	4.87	4.96	4.79
Equivalent Capacity	A.H.	1.26	1.52	1.24	1.20	1.44	1.27	1.52	1.41	1.44	1.39

Note: These plates are all 2 mm. thickness  
Average Pore Volume (approx.) 11.6 cc  
See Also Item 4, Table XXXIV

8. Appendix (cont.)

Table No. XX

## Atmospheric Impregnation of Positive Plates

## Multiple Impregnations of 4 mm. Plates

Experiment No.	Im	151A	151B	151C	151D	151E	151F	151G
Date	1947	10/2	10/3	10/3	10/3	10/6	10/6	10/8
Record	Vol. VII	26	27	28	29	30	31	32
Plate Description	S	335	344	345	352	353	354	355
Powder	P-21	-	-	-	-	-	-	P-21
Grid	8 x 8L	-	-	-	-	-	-	8 x 8L
Sinter Temp.	°F	1700	-	-	-	-	-	1700
Thickness	mm	4.29	4.29	4.32	4.32	4.32	4.29	4.27
Pore Volume	c.c.	23.83	23.80	23.98	23.91	24.02	23.83	23.71
Volume sq. in x mm		45.02	45.02	45.40	45.40	45.40	45.02	44.90
Impregnation Cycles	No.	1	2	3	4	4	4	4
Solution		Ni(NO <sub>3</sub> ) <sub>2</sub>	-	-	-	-	-	Ni(NO <sub>3</sub> ) <sub>2</sub>
Conditions		Room Temperature	-	-	-	-	-	Rm. Temp
Time	Hr.	1	1	1	1	1	1	5 min.
Pressure		Atmospheric	-	-	-	-	-	5 mm Hg
Acidity-Initial		3-4 g. HNO <sub>3</sub> /liter	-	-	-	-	-	3-4g/L
Cathodic Polarization								
Current	Amps	21	21	21	21	21	21	21
Time	Min.	36	30	32	25	30	30	32
Volts, Initial		0.97	0.99	1.00	1.16	0.95	1.01	0.96
Final		2.08	2.04	2.08	2.04	2.06	2.07	2.12
Temp., Initial	°C	93	93	93	93	94	93	93
Washing	Hrs.	3	3	3	3	3	3	3
Drying		Overnight	-	-	-	-	-	Overnight
Results								
Gain in Wt. - Total	g.	8.93	15.13	21.60	26.96	26.31	26.30	28.90
Equivalent Capacity	A.H.	2.58	4.38	6.25	7.81	7.62	7.62	8.36
Capacity Test								
Test Cell	No.	-	-	-	121	-	G-2	

Note: 151G is the Vacuum Control

8. Appendix (cont.)

Table No. XXI

## Atmospheric Impregnation of Positive Plates.

## Multiple Impregnations of 2 mm Plates.

<u>Experiment No</u>	Im	152A	152B	152C	152D	152E	152F	152G
Date	1947	10/2	10/3	10/3	10/3	10/6	10/6	10/8
<u>Record</u>	Vol. VII	P26	27	28	29	30	31	32
<u>Plate Description</u>	S	318	320	330	336	340	346	359
Powder		P-21	-	-	-	-	-	P-21
Grid		8 x 8L	-	-	-	-	-	8 x 8L
Sinter Temp.	OF	1700	-	-	-	-	-	1700
Thickness	m.m.	2.26	2.26	2.26	2.26	2.26	2.26	2.24
Pore Volume	c.c.	11.85	11.79	11.80	12.00	11.91	11.97	11.77
Volume sq. in x mm		23.78	23.78	23.78	23.78	23.78	23.78	23.60
<u>Impregnation. Cycles</u>	No.	1	2	3	4	4	4	4
Solution		Ni(NO <sub>3</sub> ) <sub>2</sub>	-	-	-	-	-	Ni(NO <sub>3</sub> ) <sub>2</sub>
Conditions		Room Temp	-	-	-	-	-	Rm. Temp
Time	Hrs.	1	1	1	1	1	1	5 min.
Pressure		Atmospheric	-	-	-	-	-	5 mm Hg
Acidity-Initial		3-4g HNO <sub>3</sub> /Liter	-	-	-	-	-	3-4g/L
<u>Cathodic Polarization</u>								
Current	Amps	21	21	21	21	21	21	21
Time	Min.	36	29	36	25	35	30	30
Volts, Initial		1.10	1.12	1.05	1.21	0.99	1.31	1.01
Final		2.26	2.08	2.14	2.25	2.28	2.19	2.16
Temp., Initial	°C	95	93	92	92	93	92	94
<u>Washing</u>	Hrs.	3	3	3	3	3	3	3
<u>Drying</u>		Overnight	-	-	-	-	-	Overnight
<u>Results</u>								
Gain in Wt - Total	g.	4.12	7.03	9.97	12.33	12.40	12.12	13.13
Equivalent Capacity	A.H.	1.19	2.03	2.89	3.58	3.59	3.51	3.80
<u>Capacity Test</u>								
Test Cell		-	-	-	96	G-1	-	-

Note: 152G is the Vacuum Control

8. Appendix (contd.)

Table No. XXII

## Atmospheric Impregnation of Positive Plates

## Effect of Temperature - 120°F

<u>Experiment No.</u>	Im	153A	153B	153C	153D	153E	153F
<u>Date</u>	1947	11/14	11/14	11/14	11/14	11/14	11/14
<u>Record</u>	Vol. VII	97	98	99	100	101	102
<u>Plate Description</u>	S	418	420	421	436	437	442
<u>Powder</u>	Type	P-21	-	-	-	-	-
<u>Grid</u>	"	8x8L	-	-	-	-	-
<u>Sinter Temp.</u>	°F	170.0	-	-	-	-	-
<u>Thickness</u>	mm	4.47	4.50	4.50	4.48	4.49	4.50
<u>Pore Volume</u>	c.c.	24.62	24.91	24.95	24.64	24.83	24.68
<u>Volume sq.in x mm.</u>		47.00	47.30	47.30	47.10	47.20	47.30
<u>Impregnation Cycles</u>		1	1	1	1	1	1
<u>Solution</u>		Ni(NO <sub>3</sub> ) <sub>2</sub>	-	-	-	-	-
<u>Conditions</u>		Constant Temp.	-	120°F	-	-	-
<u>Time</u>	Hr.	1	2	3	4	5	6
<u>Pressure</u>		Atmospheric	-	-	-	-	-
<u>Acidity-Initial</u>		3-4g. HNO <sub>3</sub> /Liter	-	-	-	-	-
<u>Cathodic Polarization</u>							
<u>Current</u>	Amps.	21	21	21	21	21	21
<u>Time</u>	Min.	30	30	34	30	35	30
<u>Volts, Initial</u>		1.59	1.63	1.61	1.59	1.50	1.68
<u>Final</u>		1.91	1.99	2.03	2.04	1.96	2.06
<u>Temp, Initial</u>	°C	92	113	98	107	108	101
<u>Washing</u>	Hr.	3	3	3	3	3	3
<u>Drying</u>		Overnight	-	-	-	-	-
<u>Results</u>							
<u>Gain in Wt-Total</u>	g.	11.80	12.69	12.32	11.71	10.77	10.95
<u>Equivalent Capacity</u>	A.H.	3.41	3.67	3.56	3.39	3.11	3.17

8. Appendix (contd.)

Table No. XXIII

## Atmospheric Impregnation of Negative Plates

Effect of Time from 1 to 6 hrs. at 120°F

Experiment No.	Im	154A	154B	154C	154D	154E	154F
Date	1947	11/14	11/14	11/14	11/14	11/14	11/14
Record	Vol. VII	97	98	99	100	101	102
Plate Description	S	388	395	397	426	430	439
Powder	Type	P-21	-	-	-	-	-
Grid	"	8x8L	-	-	-	-	-
Sinter Temp.	°F	1700	-	-	-	-	-
Thickness	mm	4.33	4.30	4.32	4.32	4.32	4.31
Pore Volume	cc	24.04	23.81	23.92	23.70	23.91	23.69
Volume sq.in.xmm		45.50	45.20	45.40	45.40	45.40	45.30
Impregnation, Cycles		1	1	1	1	1	1
Solution		Cd(NO <sub>3</sub> ) <sub>2</sub>	-	-	-	-	-
Conditions		Constant Temp.	-	120°F	-	-	-
Time	Hr.	1	2	3	4	5	6
Pressure		Atmospheric	-	-	-	-	-
Acidity, Initial		3.4g. HNO <sub>3</sub> /liter	-	-	-	-	-
Cathodic Polarization							
Current	Amps.	21	21	21	21	21	21
Time	Min.	30	30	34	30	35	30
Volts, Initial		1.66	1.57	1.64	1.69	1.81	1.68
Final		2.10	2.09	2.13	2.22	2.39	2.18
Temp, Initial	°C	92	113	98	107	108	101
Washing	Hr.	3	3	3	3	3	3
Drying		Overnight	-	-	-	-	-
Results							
Gain in Wt.-Total	g.	14.94	15.72	15.52	15.46	15.56	16.35
Equivalent Capacity	A.H.	5.47	5.76	5.69	5.66	5.70	5.98
Unit Gain							
g per cc Pore	Vol	0.622	0.660	0.650	0.652	0.652	0.690

3. Appendix (contd.)

Table No. XXIV

## Vacuum Impregnation of Negative Plates

FM-3 Plaques from 1000 lb. Batch of Powder

<u>Experiment No.</u>	<u>Date</u>	<u>Record</u>	<u>Plate Description</u>	<u>Powder</u>	<u>Grid</u>	<u>Sinter Temp.</u>	<u>Thickness</u>	<u>Pore Volume</u>	<u>Volume sq.in.x mm</u>	<u>Impregnation, Cycles</u>	<u>Solution</u>	<u>Conditions</u>	<u>Time</u>	<u>Pressure</u>	<u>Acidity-Initial</u>	<u>Cathodic Polarization</u>	<u>Current</u>	<u>Time</u>	<u>Volts, Initial</u>	<u>Final</u>	<u>Temp, Initial</u>	<u>Washing</u>	<u>Drying</u>	<u>Results</u>	<u>Gain in Wt - Total</u>	<u>Equivalent Capacity</u>
Im.	1947	Vol. IX	FM-3	Type P-23-2	Type 8x8 WL	OF	mm	cc.	No.	Min.	Amps	Min.	oc	Hrs.	g.	A.H.										
	12/16	48	163	155A	155B	155C	155D	155E	156A	156B	156C	156D	156E													
	12/16	48	164	12/16	48	12/16	48	12/16	48	12/16	48	12/16	48	12/16	48	12/16	48	12/16	48	12/16	48	12/16	48	12/16	48	12/16
	12/16	48	164	179	180	181	174	175	176	177	178															
	1715																									
	4.52	4.50	4.67	4.80	4.65	4.60	4.47	4.55	4.72	4.47																
	31.54	31.53	33.21	34.00	33.04	32.07	31.91	32.22	32.90	31.83																
	62.40	62.60	65.40	68.40	65.30	64.00	62.50	63.50	65.60	62.60																
	3	3	3	3	3	4	4	4	4	4																
	Cd(NO <sub>3</sub> ) <sub>2</sub>																									
	Room Temperature																									
	5	5	5	5	5	5	5	5	5	5																
	6-8 mm. Hg.																									
	3-4g. HNO <sub>3</sub> /liter																									
	28	28	28	28	28	28	28	28	28	28																
	25-30																									
	1.10																									
	2.00																									
	104	104	104	104	104	104	107	107	107	107																
	3	3	3	3	3	3	3	3	3	3																
	Overnight																									
	49.47	49.13	52.02	53.52	53.59	60.59	60.50	59.36	58.61	55.86																
	18.10	17.98	19.07	19.61	19.68	22.19	22.16	21.73	21.47	20.43																

8. Appendix (cont.)

Table No. XXV

Vacuum Impregnation of Positive Plates

FM-3 Plaques from 1000 lb batch of Powder

<u>Experiment No.</u>	IM	157A	157B	157C	157D	157E
Date	1947	12/15	12/15	12/15	12/15	12/15
<u>Record</u>	Vol. IX	46	46	46	46	46
<u>Plate Description</u>	FM-3	169	170	171	172	173
Powder	Type	P-23-2	-	-	-	-
Grid	Type	8 x 8L	-	-	-	-
Sinter Temp.	°F	1715	-	-	-	-
Thickness	m.m.	4.50	4.74	4.67	4.52	4.57
Pore Volume	c.c.	31.53	32.66	32.39	32.54	31.98
Volume, sq.in.x mm		62.10	65.30	64.50	63.80	63.60
<u>Impregnation, Cycles</u>	No.	4	4	4	4	4
Solution		Ni(NO <sub>3</sub> ) <sub>2</sub>	-	-	-	-
Conditions		Room Temperature	-	-	-	-
Time	Min.	5	5	5	5	5
Pressure		5-8 mm Hg.	-	-	-	-
Acidity-Initial		3-4 g HNO <sub>3</sub> /Liter	-	-	-	-
<u>Cathodic Polarization</u>						
Current	Amps	28	28	28	28	28
Time	Min.	25-30	-	-	-	-
Volts, Initial		1.20	1.20	1.20	1.20	1.20
Final		2.10	2.10	2.10	2.10	2.10
Temp, Initial	°C	106	106	106	106	106
<u>Washing</u>	Hrs.	3	3	3	3	3
<u>Drying</u>		Overnight	-	-	-	-
<u>Results</u>						
Gain in Wt - Total	g.	36.04	38.85	38.53	36.76	38.26
Equivalent Capacity	A.H.	10.40	11.20	11.10	10.60	11.10

8. Appendix (cont.)

Table No. XXVI

Vacuum Impregnation of Plates

Positive and Negative S Plates

Expt No Im	Plate Nos S	Plate	Cycles of	Average Gain	Theoretical		
		Thick mm.	Impregnation No	in Weight Total g	Capacity	A.H.	
Av. Max. Min.							
<u>Impregnation to Produce Positive Plates</u>							
165	399,401,402, 404,414	2	3	9.52	2.75	2.96	2.33
166	342,390,396, 398,408	4	3	22.11	6.40	6.76	6.21
167	400,394,384, 385,386	2	4	12.94	3.76	3.79	3.63
168	343,351,434 435,369	4	4	27.99	8.09	8.92	7.67

Impregnation to Produce Negative Plates

169	337,338,347 356,358	2	3	15.82	5.80	5.95	5.78
170	409,427,428 431,433	4	3	38.83	14.15	14.53	13.21
171	317,326,327 328,329	2	4	19.35	7.08	7.42	6.95
172	410,415,416 417,419	4	4	45.36	16.56	17.78	15.81

Notes (1) Record Vol VII, pp. 103-6; 11/17-11/20 (1947)  
 (2) All impregnations and polarizations, including washing  
 and drying under usual standard conditions for  
 vacuum impregnation.



## 8. Appendix (contd.)

Table No. XXVII

Vacuum Impregnation of Positive Plates  
Effect of Sintering Time for Plaques

Experiment No.	Im	158A	158B	158C	158D	158E	158F	158G	158H	158I	158J	158K
Date	1947	11/25	11/25	11/25	11/25	11/25	12/2	12/2	12/2	12/2	12/2	12/2
Record	Vol. IX	22	22	22	22	22	29	29	29	29	29	29
Plate Description	FM-3	152	153	154	155	156	157	158	159	160	161	162
Powder	Type	P-21-2										
Grid	8x8											
Sinter Temp. & Time	of 1800	5Min	6Min	7Min	8Min	9Min	10Min	11Min	12Min	13Min	14Min	15Min
Thickness	mm	5.94	5.79	5.66	5.56	5.41	5.35	5.23	5.28	5.05	4.86	4.90
Pore Volume	cc	43.95	42.61	41.49	40.81	39.31	38.22	37.06	37.24	35.48	34.00	33.86
Volume sq. in x mm		83.30	82.00	80.10	79.00	76.40	74.60	72.50	73.30	70.50	68.00	67.70
Impregnation, Cycles	No.	4	4	4	4	4	4	4	4	4	4	4
Solution		Ni(NO <sub>3</sub> ) <sub>2</sub>										
Conditions		All at Room Temperature										
Time	Min.	5	5	5	5	5	5	5	5	5	5	5
Pressure	mm Hg.	5 to 9										
Acidity-Initial		3-4 g. HNO <sub>3</sub> /liter										
Cathodic Polarization												
Current	Amps.	26	26	26	26	26	26	26	26	26	26	26
Time	Min.	25-35										
Volts, Initial		1.10	1.10	1.10	1.10	1.10	1.3	1.30	1.30	1.30	1.30	1.30
Final		2.20	2.20	2.20	2.20	2.20	2.10	2.10	2.10	2.10	2.10	2.10
Temp., Initial	°C	107	107	107	107	107	105	105	105	105	105	105
Washing	Hrs.	3	3	3	3	3	3	3	3	3	3	3
Drying		Overnight										
Results												
Gain in Wt - Total	g.	55.40	53.13	52.28	49.64	49.08	49.22	48.42	47.32	45.51	43.81	43.65
Equivalent Capacity	A.H.	16.02	15.37	15.11	14.35	14.20	14.24	14.00	13.68	13.16	12.67	12.62
Unit Gain	*	1.26	1.25	1.26	1.22	1.25	1.29	1.31	1.27	1.28	1.29	1.29
Capacity Test												
Test Cell	No.	241	242	243	244	245	246	247	248	249	250	251

Note \* Expressed in grams (Ni(OH)<sub>2</sub> per cc of pore volume, calculated from porosity of plaque.

# 8. Appendix (cont.)

Table No. XXVIII

## Vacuum Impregnation of Positive Plates

### FM-1, FM-2, and FM-3 Plaques.

Expt No IM	Plate Type	Description Plate N. s	Number of Plates	Average Gain in weight Total g.	Theoretical Capacity -- A.H.		
					Average	Max.	Min.
162	FM-1	3-21	18	29.57	8.55	9.01	8.11
163	FM-2	2-19	18	33.99	9.83	10.74	8.70
164	FM-3	3-114	104	37.81	10.91	12.44	9.06
164	"	*	10	39.99	11.56	11.93	11.22
164	"	**	5	31.84	9.23	9.41	8.78

Notes (1) For description of plates, see Tables VII, VIII, and IX.

(2) All plates were impregnated for 5 minutes in vacuum in nickel nitrate solution at room temperature. The cycle was repeated four times, except as noted below. Cathodic polarisation was under the usual conditions with the 25% KOH solution heated to an initial temperature of 90-100°C. Current density averaged 1 amp. per sq. in., counting both sides of the plate. All plates were washed in cold running water for 3 hours and dried overnight.

(3) Beginning with the FM-2 plates, the 5 cell electrolysis apparatus was used and the number of plates impregnated in one step was increased to five.

(4) Record, Vol. VII 38-86, IX 4-6.

\*These plates impregnated five times.

\*\* " " " three times only.

8. Appendix (contd.)

Table No. XXIX

		Vacuum Impregnation of Plates									
		Cathodic Polarization in Lithium Hydroxide Solution									
Experiment No.	Im	159A	159B	159C	159D	159E					
Date	1947	11/24	11/24	11/24	11/24	11/24					
Record	Vol. IX	20&27	20&27	20&27	20&27	20&27					
Plate Description	S	348	357	360	339	350					
Powder	Type	P-21									
Grid	"	8x8L									
Sinter Temp.	°F	1700									
Thickness	mm	2.3	2.2	2.0	2.0	2.3					
Pore Volume	cc	12.18	11.48	10.48	10.39	12.35					
Volume sq.in.xmm		24.19	23.14	21.02	21.02	24.19					
Impregnation, Cycles	No.	4	4	4	4	4					
Solution		Ni(NO <sub>3</sub> ) <sub>2</sub>	Ni(NO <sub>3</sub> ) <sub>2</sub>	Ni(NO <sub>3</sub> ) <sub>2</sub>	Cd(NO <sub>3</sub> ) <sub>2</sub>	Cd(NO <sub>3</sub> ) <sub>2</sub>					
Conditions		All at Room Temperature									
Time	Min.	5	5	5	5	5					
Pressure	mm Hg.	7.5-10.0									
Acidity-Initial		From 3.4 g/L HNO <sub>2</sub>									
Cathodic Polarization		LiOH solution, 150 g LiOH per L									
Current	Amps.	21	21	21	21	21					
Time	Min.	From 20-31 Minutes									
Volts, Initial		From 1.9-2.2									
Final		From 2.4-2.5									
Temp., Initial	°C	98 - 103									
Washing	Hr.	3	3	3	3	3					
Drying		Overnight									
Results											
Gain in Wt.-Total	g.	14.18	14.50	12.91	19.63	21.86					
Equivalent Capacity	A.H.	4.09	4.34	3.73	7.19	8.02					

8. Appendix (cont.)

Table No. XXX

Vacuum Impregnation of Porous Graphite Plates

Experiment No.	Im	161A	161B	161C	161D	161E	161F
Date	1947	12/29	-	-	-	-	-
Record	Vol.IX	49-50	-	-	-	-	-
Operator		R.R.	-	-	-	-	-
<u>Plate Description</u>	No	A-12-1	50-1	40-1	D-30-1	E-12-1	E-20-1
		National Carbon Co porous graphite plates, machined to size, 4 x 3 x 0.25 inches from standard stock.					
Pore Diameter	micron	33	48	69	99	140	140
Pore Volume *	cc	24.51	24.96	25.66	26.00	26.96	26.96
Designated Grade	No	60	50	40	30	20	20
<u>Impregnation, Cycles</u>	No	4	-	-	-	-	-
<u>Solution</u>		Ni Nitrate					
<u>Conditions</u>		Room Temperature, 6 - 9 mm Hg Pressure					
Time	Min	5	-	-	-	-	-
Acidity		3 - 4 g HNO <sub>3</sub> per L					
<u>Cathodic Polarization</u>							
Current	amps	24	-	-	-	-	-
Time	min	30	-	-	-	-	-
Volts, Initial		1.7	-	-	-	-	-
Final		2.2	-	-	-	-	-
Temp., Initial	°C	106	-	-	-	-	-
<u>Washing</u>	hrs	3	-	-	-	-	-
<u>Drying</u>		Overnight					
<u>Results</u>							
Gain in Weight							
1st Impregnation	g	7.10	6.27	5.78	5.54	6.03	5.03
2nd "	g	5.97	7.81	5.78	4.99	5.07	4.91
3rd "	g	3.38	6.83	7.67	6.26	4.89	5.06
4th "	g	1.58	7.49	4.82	5.31	6.12	4.55
Total	g	18.03	28.40	24.05	22.10	22.11	19.55
Unit gain							
g per cc of Pores		0.736	1.138	0.938	0.851	0.820	0.727
% by volume		18.2	27.7	23.1	20.7	20.0	17.8

Note \* Calculated, assuming density of graphite to be 2.20

8. Appendix (cont.)

Table No. XXXI

Atmospheric Impregnation of Positive Plates

Effect of Immersion for 1 and 2 days

<u>Experiment No</u>	Im	160A	160B	160C	160D
Date	1947	12/17	12/17	12/18	12/18
<u>Record</u>	Vol. IX	50	50	50	50
<u>Plate Description</u>	FM-3	165	166	167	168
Powder	Type	P-23-2	P-23-2	P-23-2	P-23-2
Grid	"	8 x 8L	8 x 8L	8 x 8L	8 x 8L
Sinter Temp.	°F	1715	1715	1715	1715
Thickness	mm	4.47	4.50	4.42	4.34
Pore Volume	cc	31.90	31.85	31.28	30.56
Volume sq.in.x mm		62.60	62.70	61.70	60.50
<u>Impregnation, Cycles</u>	No.	1	1	1	1
Solution		Ni(NO <sub>3</sub> ) <sub>2</sub>	Ni(NO <sub>3</sub> ) <sub>2</sub>	Ni(NO <sub>3</sub> ) <sub>2</sub>	Ni(NO <sub>3</sub> ) <sub>2</sub>
Conditions		Room Temperature	Room Temperature	Room Temperature	Room Temperature
Time	Hr.	24	24	48	48
Pressure		Atmospheric	Atmospheric	Atmospheric	Atmospheric
Acidity-Initial		3-4g/HNO <sub>3</sub> /L	3-4g/HNO <sub>3</sub> /L	3-4g/HNO <sub>3</sub> /L	3-4g/HNO <sub>3</sub> /L
<u>Cathodic Polarization</u>					
Current	Amps	28	28	28	28
Time	Min.	30	30	25	25
Volts, Initial		1.85	1.85	1.90	1.90
Final		2.10	2.10	2.10	2.10
Temp., Initial	°C	103	103	104	104
<u>Washing</u>	Hrs.	3	3	3	3
<u>Drying</u>		Overnight	Overnight	Overnight	Overnight
<u>Results</u>					
Gain in Wt - Total	g.	22.25	22.58	20.43	25.85
Equivalent Capacity	A.H.	6.44	6.53	5.91	7.48

8. Appendix (cont.)

Table No. XXXII

Drying of Positive Plates

Variation with Cycles of Impregnation

Plate No.	Impregnation Data	Cycle No.	Water	Ni(OH) <sub>2</sub>	Pore Volume	
			Retained (g) or (cc) (Note #1)	Vol. c.c. (Note #2)	Found c.c. (Note #3)	Calc'd c.c.
FM-3 169	5 Min. Vac, Pos, 4x	1	27.0	2.82	29.82	31.52
		2	24.2	5.15	29.35	31.52
		3	22.9	7.63	30.53	31.52
		4	20.2	8.80	29.00	31.52
170	"	1	27.9	3.11	31.01	32.66
		2	25.8	5.58	31.38	32.66
		3	23.4	8.13	31.53	32.66
		4	21.4	9.48	30.88	32.66
171	"	1	27.4	2.99	30.39	32.39
		2	24.8	5.50	30.30	32.39
		3	24.2	7.88	32.08	32.39
		4	20.8	9.40	30.20	32.39
172	"	1	26.9	2.88	29.78	32.54
		2	24.8	5.25	30.05	32.54
		3	22.6	7.68	30.28	32.54
		4	20.3	8.96	29.26	32.54
173	"	1	26.6	2.98	29.58	31.98
		2	25.2	5.36	30.56	31.98
		3	23.5	7.80	31.30	31.98
		4	20.8	9.34	30.14	31.98

Note #1 Difference in weight of plate upon removal from washing process, and upon removal from drying cabinet.

Note #2 Calculated from net gain in weight of plate, using density of Ni(OH)<sub>2</sub> as 4.10 g/cc.

Note #3 Sum of columns 1 and 2.

8. Appendix (contd.)

Table No. XXXIII

Drying of Negative Plates

Variation with Cycles of Impregnation

Plate No.	Impregnation Data	Cycle, No.	Water Retained	Ni(OH) <sub>2</sub> Vol	Pore Volume	
					Found	Calc'd
			(g) or (cc)	c.c.	c.c.	c.c.
			(Note #1)	(Note #2)	(Note #3)	
FM3	5 Min. Vac, Neg. 4x	1	26.7	3.89	30.59	32.07
174		2	23.6	7.52	31.12	32.07
		3	19.8	10.47	30.27	32.07
		4	16.4	12.64	29.04	32.07
175	"	1	27.3	3.76	31.06	31.91
		2	23.1	7.33	30.43	31.91
		3	19.5	10.37	29.87	31.91
		4	15.5	12.63	28.13	31.91
176	"	1	26.7	3.83	30.53	32.22
		2	23.8	7.49	31.29	32.22
		3	19.8	10.63	30.43	32.22
		4	16.2	12.38	28.58	32.22
177	"	1	27.8	3.89	31.69	32.90
		2	23.9	7.63	31.53	32.90
		3	20.0	10.80	30.80	32.90
		4	16.9	12.22	29.12	32.90
178	"	1	27.0	3.53	30.53	31.83
		2	23.5	7.05	30.55	31.83
		3	19.6	10.11	29.71	31.83
		4	16.8	11.64	28.44	31.83

Note #1 Difference in weight of plate upon removal from washing process, and upon removal from drying cabinet.

Note #2 Calculated from net-gain in weight of plate, using density of Cd(OH)<sub>2</sub> as 4.79 g/c.c.

Note #3 Sum of columns 1 and 2.

## 8. Appendix (Conts.)

### Table No. XXXIV

#### Notes to Tables XIV to XXXIII on Impregnation.

1. Atmospheric Impregnation. The impregnation process covers the steps of immersing a plaque in a nickel or cadmium salt solution, cathodic polarization in caustic solution, washing and drying. These steps constitute a cycle of the impregnation process which introduces nickelous or cadmium hydroxide into the plaque. The atmospheric impregnation refers to the immersion of the plaque in a nickel or cadmium salt solution and maintaining it in the solution for a specified period. In the vacuum process, on the other hand, the plaque is first placed in a suitable vacuum vessel, the system is evacuated to about 5 or 6 mm. Hg pressure, and the nickel or cadmium salt solution is introduced. The system is maintained at the vapor pressure of the solution for the desired period. The volume of solution for the atmospheric impregnations varied from 0.5 to 1 liter in comparison to a volume of 1.5 liters in the vacuum impregnations.
2. Electrolysis Apparatus. To permit a speeding up of the experiments on impregnation, a new apparatus was set up permitting the simultaneous electrolysis of five plates in one operation. The apparatus consisted of five nickel cans connected in series with the source of d.c. current. The cans were set into a suitable supporting tray, which was placed next to a nickel can in which the caustic electrolyte could be heated to the desired temperature and which could be tilted to fill the five electrolysis cans simultaneously. The procedure was to transfer the plates from the impregnation apparatus to the individual nickel cans as quickly as possible, making the electrical connections, and then filling with electrolyte from the tilting reservoir. This unit was put into operation on October 23rd, and a second unit added when the first one was found to be convenient and satisfactory for this process.
3. Plaques. All plaques used in the experiments reported herein, were made from light carbonyl nickel powder as described in the Discussion under Sintering, except for those in Table No. XXX which were porous graphite plaques obtained from National Carbon Co.



8. Appendix (Contd.) Table XXXIV (contd.)

4. Impregnation at Constant Acidity. Table XIX. Expt IM 149. The amount of acid consumed during the impregnation process was estimated from a trial curve showing the change in acid content with time. For this experiment, five plates were immersed in the nickel nitrate solution in the bowl of a desiccator, provided with an agitator, and a nickel nitrate-nitric acid solution containing 100 g  $\text{HNO}_3$  per liter added from a burette. Since the trial curve showed a greater rate of consumption during the initial portion of the immersion followed by a slow consumption and then by a second period of increased consumption, the schedule of adding the acid nickel nitrate solution was made as follows:

During 1st 10 minutes	25 ml
" middle 40 minutes	25 "
" last 10 minutes	22.4 ml.

This experiment showed an increase in acidity over the average initial acidity. In Experiment IM 150, the acid nickel nitrate solution was added at a steady rate of about 1 ml per minute. Acidity was checked at ten minute intervals.

8. Appendix (contd.)

Table No. XXXV

Test of Cells with Pocket Type Positive and Negative Plates

Test Rated Capacity			Electrolyte	Cycles	Capacity		
Cell Positive		Negative			A.H.		
No.	A.H.	A.H.	KOH, d 1.20, Plus	No	Average	High	Low
T							
87	2	8	K <sub>2</sub> SO <sub>4</sub> (satd)	22	2.27	2.67	1.83
88	4	4	17.5 g/L	22	4.49	5.41	3.67
89	2	8	KCl	22	2.27	2.67	1.83
90	4	4	15.0 g/L	22	4.38	5.25	3.58
91	2	8	K <sub>2</sub> CO <sub>3</sub>	22	2.27	2.67	1.83
92	4	4	14.0 g/L	22	4.01	5.00	3.17
93	4	4	Standard	29	4.42	4.57	3.67
110	8	4	Standard	29	6.03	7.17	3.83
123	8	8	Standard	8	7.46	8.33	6.08
106	8	4	"	20	6.03	7.25	4.25
"	"	"	"	*36	5.64	7.25	4.25
107	2	16	"	20	2.31	2.67	1.83
"	"	"	"	*36	2.18	2.67	1.75

Note \* includes first 20 cycles, immediately above.

8. Appendix (contd.)

Table No. XXXVI

Test Cells of Vacuum Impregnated Positive FM-3 Plates

Test Cell No	T					98	99	125
<u>Impregnation Data</u>								
Table No						XXVIII	-	-
Experiment No	Im					164	-	-
Plate No	FM-3					22	23	24
Plate Thickness	mm					4.50	4.34	4.37
Conditions								
Gain in Weight	g					38.84	36.89	37.24
Theoretical Capacity	A.H.					11.23	10.67	10.77
<u>Test Cell Assembly</u>								
Rated Capacity	A.H.					9.2	8	16
Testing Results								
Cycle No	Date	Current	Duration	Input	Time	Current	*Elapsed Discharge Capacity	
							to 1.0 Volt	
	1947	Amps	hrs	A.H.	Hrs.	Amps	A.H.	A.H.
1	11/5-11/7	1	39	39	0	2-2.5	9.61	8.52
2	11/7-11/12	1	19	19	96	2	8.86	8.12
3	11/12-11/13	2	15	30	2.5	3	9.69	8.59
4	11/13-11/14	2	15	30	5	4	9.66	8.40
5	11/14-11/17	0.5	63	31.5	0.25	2	9.50	8.82
6	11/17-11/18	1.5-2	8.5	14.5	7	2	8.94	8.16
7	11/19	2	7	14.0	0.5	2	9.50	8.54
8	11/20	2	7	"	"	"	9.10	8.32
9	11/21	"	"	"	"	3	9.00	8.25
10	11/24	"	"	"	16	4	8.68	7.68
11	11/26	"	"	"	0	4	9.48	8.44
12	12/3	"	"	"	0.5	4	8.68	8.00
13	12/4	"	"	"	"	10	5.44	1.33
14	12/5	"	"	"	"	10	5.00	1.67
15	12/9	1.5	"	10.5	"	2	8.26	7.66
16	12/10	"	"	"	"	2	8.34	7.94
17	12/12	2	"	14.0	"	2	8.87	8.40

Note (\*) Elapsed Time indicates time between end of charging and start of discharge.

8. Appendix (contd.)

Table No. XXXVII

Test Cells of Vacuum Impregnated Negative Plates

Test Cell No	T	101	102	103	104	105	86	109
<u>Impregnation Data</u>								
Table No - 9/30/47 Report		XXI	-	-	-	-	-	XXII
Experiment No	Im	82	83	87	88	90	115	116
Plate No	S	235	231	243	223	245	250	251
Plate Thickness	mm	2.26	2.21	2.33	2.15	2.26	2.24	2.30
Conditions		Vacuum - 5 minutes						
Solution		Cd Chloride						
Temperature		Room						
No. of Cycles		1	2	2	3	4	1	-
Gain in Weight	g	5.2	8.8	9.2	12.7	18.5	5.81	6.31
Theoretical Capacity	A.H.	1.90	3.22	3.36	4.65	6.78	2.13	2.31
<u>Test Cell Assembly</u>								
Rated Capacity	A.H.	8	-	-	-	-	4	8
<u>Testing Results</u>								
No of Cycles		29	-	-	-	-	22	-
Capacity								
Average	A.H.	1.27	2.45	2.48	3.44	5.02	1.74	1.83
High	A.H.	1.58	2.75	2.83	4.00	5.83	2.17	2.25
Low	A.H.	1.00	2.17	2.25	3.00	4.58	1.58	1.58
Coeff of Utilisation	%	66.9	76.1	73.8	74.0	74.1	81.7	79.3

Table No. XXXVIII

## Test Cells, including all-sintered plate Batteries

Test Cell No.	T	85	108	115	74-105	86-18-50
<u>Impregnation Data</u>		<u>Positive Plate Only</u> - - - - -				
Table No.		9-30-47, No. XVII, XVI, XIX 6-30-47, XXXV				
Experiment No.	Im	117	118	62	96	23
Plate Thickness	mm.	4.28	- - -	4.73	4.28 -	*(4) 4.27
Plate No.	S	310	311	184	267	57
<u>Conditions</u>						
Temperature	°C	28-25		Room	99	20
Pressure		Vacuum - - - - -			Atm	Atm
Time	min	5	5	5	30	20 hrs
No. of Cycles		1	1	10	1	1
Gain in Weight	g	7.34	7.22	50.4	16.44	13.5
Theoretical Capacity	A.H.	2.12	2.09	14.6	4.76	3.91
<u>Testing Cell Assembly</u>						
Pocket Negatives						
Rated Capacity	A.H.	18	16	16	- - -	- - -
Sintered Negatives	No				S245	S187 S250
Theor. Capacity	A.H.	- - -	- - -	- - -	6.78	5.81 2.13
<u>Testing Results</u>						
No. of Cycles		22	22	19	19	19
Capacity						
Average	A.H.	1.56	1.56	8.92	3.76	5.16
High	A.H.	2.00	2.00	11.33	4.08	5.75
Low	A.H.	1.33	1.33	6.67	3.50	4.64
Coeff. of Utilisation	%	73.6	74.7	61.1	78.8	132

Notes (1) T 74-105 has the positive plate from T 74 combined with the negative plate of T 105.

(2) T 86-18-50 has positive plate of T 18 combined with negative plates from T 50 and T 86.

(3) All plates in T 74-105 & T 86-18-50 had been cycled previously.

(4) This plate was made from Ineo #2, heavy powder.

8. Appendix (contd.)

Table No. XXXIX

Testing of Capacity of Cells

Effect of Sintering Time in Making Plaque

Test Cell No.	T	80	81	82	83	84
<u>Plaque Data</u>						
Plaque No.	S	218	219	220	221	222
Sinter Time	min	10	15	20	25	30
<u>Impregnation Data</u>						
Table No. - 9/30/47	Report	XVIII	-	-	-	-
Plate Thickness	mm.	2.19	2.03	1.96	1.84	1.85
Conditions		Time, 5 minutes	-	-	-	-
Solution		50% Ni Nitrate	-	-	-	-
Temperature		Room	-	-	-	-
Pressure		Vacuum	-	-	-	-
No. of Cycles		3	-	-	-	-
Gain in wt.	g	11.1	9.5	9.1	8.5	8.7
Theoretical Capacity	A.H.	3.21	2.74	2.63	2.46	2.51
<u>Test Cell Assembly</u>						
Rated Capacity	A.H.	8	-	-	-	-
<u>Testing Results</u>						
No. of Cycles		22	-	-	-	-
<u>Capacity</u>						
Average	A.H.	2.71	2.47	2.25	1.99	2.05
High	A.H.	3.25	3.00	2.75	2.50	2.50
Low	A.H.	2.50	2.25	2.00	1.83	1.83
Coeff of Utilisation	%	84.7	90.2	85.6	80.8	81.7

Table No. XXXX

## Testing of Positive Plate Capacity

## Effect of Atmospheric Impregnation at Elevated Temperatures

Test Cell No.	T	71	72	73	74	75	111	76	112	77	113	78	114
Impregnation Data													
Table No-9/30-47 Report	XX	-	-	-	-	-	-	-	-	-	-	-	-
Experiment No.	106	112	114	301	267	268	98	-	102	-	104	-	-
Plate No.	291	300	301	301	267	268	268	-	271	-	277	-	-
Plate Thickness	4.28	-	-	-	-	-	-	-	-	-	-	-	-
Conditions	Single Cycle	-	-	-	-	-	-	-	-	-	-	-	-
Solution, % Ni(NO <sub>3</sub> ) <sub>2</sub>	50	-	-	-	60	-	-	-	-	-	-	-	-
Temperature	99	-	-	-	-	-	-	-	-	-	-	-	-
Pressure	Atmospheric	-	-	-	-	-	-	-	-	-	-	-	-
Time	0.5	3	6	6	0.5	1.0	-	-	3.0	-	6.0	-	-
Gain in Weight	10.32	13.46	12.98	16.44	15.50	19.63	-	-	21.16	-	22.90	-	-
Theoretical Capacity	2.99	3.89	3.76	4.76	5.65	5.69	-	-	6.11	-	6.63	-	-
Test Cell Assembly													
Pocket Negatives	A.H.	8	-	-	-	-	16	8	16	8	16	8	16
Rated Capacity													
Testing Results													
No. of Cycles	22	-	-	-	-	-	18	22	18	22	18	22	18
Capacity													
Average	A.H.	2.48	3.36	3.11	3.98	4.63	4.40	4.85	4.63	4.98	4.64	5.45	5.15
High	A.H.	2.83	4.08	3.67	4.83	5.75	4.58	6.08	5.00	6.17	5.08	6.50	5.75
Low	A.H.	2.33	3.00	2.83	3.58	4.00	4.08	4.17	4.25	4.25	4.25	4.58	4.75
Coeff. of Utilisation	%	83.0	86.4	82.7	83.6	82.0	77.9	85.3	81.4	81.5	75.8	82.2	77.7

8. Appendix (contd.)

Table No. XXXXI

Testing of Positive Plate Capacity

Effect of Atmospheric Impregnation at Elevated Temperatures

Test Cell No.	T	64	65	66	67	68
<u>Impregnation Data</u>						
Table No. - 9/30/47 Report		XIX - - - - -			XX - - - - -	
Experiment No.		95	97	99	107	109
Plate No.	S	262	263	264	283	284
Plate Thickness	mm	2.19	2.19	2.19	2.19	2.19
<u>Conditions</u>						
Solution, % Ni(NO <sub>3</sub> ) <sub>2</sub>		60 - - - - -			50 - - - - -	
Temperature	°C	99° - - - - -				
Pressure		Atmospheric - - - - -				
No. of Cycles		1 - - - - -				
Gain in Weight	g	8.17	9.21	11.16	5.24	6.58
Theoretical Capacity	A.H.	2.36	2.66	3.23	1.51	1.90
<u>Test Cell Assembly</u>						
Pocket Negatives						
Rated Capacity	A.H.	8 - - - - -				
<u>Testing Results</u>						
No. of Cycles		22 - - - - -				
<u>Capacity</u>						
Average	A.H.	1.87	1.96	2.74	1.14	1.69
High	A.H.	2.08	2.08	3.25	1.33	2.08
Low	A.H.	1.58	1.58	2.33	1.00	1.50
Coeff. of Utilisation	%	79.3	73.7	84.9	75.6	89.0



Table No. XXXXII

## Testing of Positive Plate Capacity

## Effect of Multiple Cycles of Atmospheric Impregnation

Test Cell No.	T	96	G-1	95	121	G-2	122
<u>Impregnation Data</u>							
Table No.		XXI	- - - - -	- - - - -	XX	- - - - -	- - - - -
Experiment No.	Im	152D	152E	152G	151D	151F	151G
Plate No.	S	336	340	359	352	354	355
Plate Thickness	mm	2.26	2.26	2.24	4.32	4.29	4.29
<u>Conditions</u>							
Temperature		Room - - - - -					
Pressure		Atmospheric Vac Atmospheric Vac					
No. of Cycles		4	- - - - -	- - - - -	- - - - -	- - - - -	- - - - -
Gain in Weight	g	12.33	12.40	13.13	26.96	26.30	28.90
Theoretical Capacity	A.H.	3.58	3.59	3.80	7.81	7.62	8.36
<u>Test Cell Assembly</u>							
Pocket Negative							
Rated Capacity	A.H.	4	- -	4	8	- -	8
Sintered Negative	Plate No.	- -	292	- -	- -	321	- -
Theoretical Capacity	A.H.	- -	6.06	- -	- -	12.58	- -
<u>Testing Results</u>							
No. of Cycles		18	- - - - -	- - - - -	- - - - -	19	18
Capacity							
Average	A.H.	2.99	2.95	3.13	6.54	6.27	6.70
High	A.H.	3.17	3.33	3.33	7.08	7.00	7.33
Low	A.H.	2.70	2.68	2.85	6.12	5.71	5.83
Coeff of Utilisation	%	83.8	82.2	82.4	83.9	82.3	80.1

Table No. XXXXLIII

## Testing of Negative Plate Capacity

## Effect of Multiple Cycles of Atmospheric Impregnation

Test Cell No.		T94	T117	T116	T118	T119	T120
<u>Impregnation Data</u>							
Table No.		XIV - - - - -			XV - - - - -		
Experiment No.		141	143	133	146	144	134
Plate No.	S	305	306	295	333	332	322
Plate Thickness	mm	2.0	2.2	2.1	4.0	4.3	4.1
<u>Conditions</u>							
Temperature		Room - - - - -					
Pressure		Atmospheric - - - - -					
Cycles	No.	2	2	3	2	2	3
Gain in Weight	g	12.58	12.40	17.02	26.57	26.59	34.8
Theoretical Capacity	A.H.	4.61	4.54	6.24	9.74	9.74	12.7
<u>Test Cell Assembly</u>							
Pocket Positives							
Rated Capacity	A.H.	4	8	8	8	9	9
<u>Testing Results</u>							
No. of Cycles		18	18	17	18	17	18
<u>Capacity</u>							
Average	A.H.	4.16	4.34	5.25	6.46	6.35	6.83
High	A.H.	4.58	4.75	6.00	8.08	7.83	9.08
Low	A.H.	3.70	3.75	4.93	5.08	5.04	5.08
Coeff of Utilisation	%	90.3	95.5	84.3	66.5	65.3	53.5